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SYNERGISTIC MECHANISMS OF SOLID LUBRICANTS.(U)
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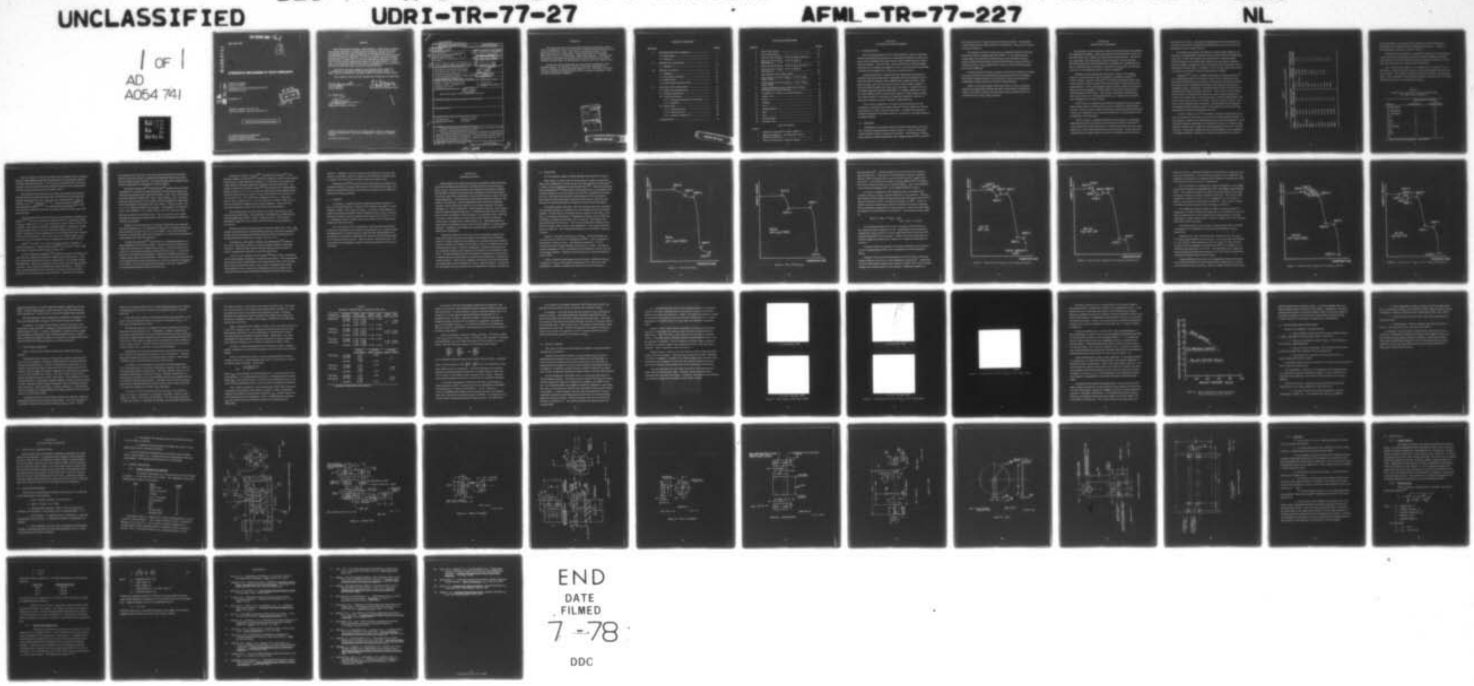

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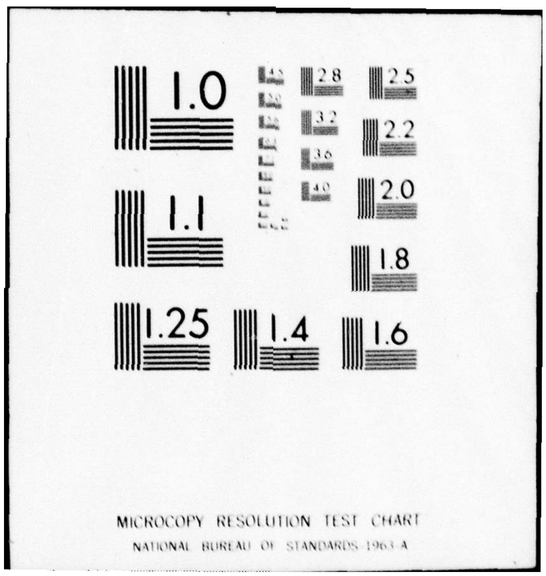
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SYNERGISTIC MECHANISMS OF SOLID LUBRICANTS

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DECEMBER 1977

TECHNICAL REPORT AFML-TR-77-227
Final Technical Report — April 1976 - September 1977

Approved for public release; distribution unlimited.

AIR FORCE MATERIALS LABORATORY
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AIR FORCE/56780/10 May 1978 — 200

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SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

19 REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER 18 AFML-TR-77-227	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) 6 SYNERGISTIC MECHANISMS OF SOLID LUBRICANTS		5. TYPE OF REPORT & PERIOD COVERED 9 Final Technical Report. 1 Apr 76 - 30 Sep 77
		6. PERFORMING ORG. REPORT NUMBER 14 UDRI-TR-77-27
7. AUTHOR(s) 10 Richard S. Harmer and Carlo G. Pantano		8. CONTRACT OR GRANT NUMBER(s) 15 F33615-76-C-5160
9. PERFORMING ORGANIZATION NAME AND ADDRESS University of Dayton Research Institute 300 College Park, Dayton, Ohio 45469		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 16 7343-02 17
11. CONTROLLING OFFICE NAME AND ADDRESS Air Force Materials Laboratory (AFML/MBT) Air Force Systems Command Wright-Patterson Air Force Base, OH 45433		12. REPORT DATE 11 December 1977
		13. NUMBER OF PAGES 53
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office) ONRRR, The Ohio State University Research Center, 1314 Kinnear Rd. Columbus, OH 43212		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

12 58p.

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

solid lubricants
molybdenum disulfide
antimony trioxide
synergism

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

Literature pertaining to synergistic enhancement of solid film lubricants by additions of non-lubricant phases is reviewed.

Research directed toward identification of mechanisms by which Sb_2O_3 additives enhance MoS_2 lubrication is described. Chemical, mechanical, and tribochemical reactions are investigated. Preliminary data indicates Sb_2O_3 alters the oxidation behavior of MoS_2. An experimental test apparatus for solid lubricant evaluation is described.

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PREFACE

This report describes work performed during the period from 1 April 1976 to 30 September 1977 by the University of Dayton Research Institute under Contract F33615-76-C-5160. This work is sponsored by the Air Force Materials Laboratory, AFML/MBT, Wright-Patterson Air Force Base, Ohio, 45433. Mr. B. D. McConnell is the Project Engineer. The work was conducted in the Metals and Ceramics Division of the University of Dayton Research Institute under the administrative supervision of Dr. Alden E. Ray.

University of Dayton personnel who made major contributions to the program include: Dr. Richard S. Harmer, Principal Investigator; Dr. Carlo G. Pantano, Research Materials Engineer; Dr. John Crisp, Associate Professor of Mechanical Engineering; and Mr. Joseph B. Meierdirks, S.M., Assistant Metallurgical Engineer.

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INTRODUCTION AND SUMMARY

1.1 INTRODUCTION

Since the announcement in 1965 of the synergistic effects of antimony trioxide on the lubricant properties of molybdenum disulfide solid film lubricants, little development has been directed toward identification of superior lubricant systems. Instead, MoS_2 - Sb_2O_3 mixtures have been thoroughly exploited from an engineering approach, that is, work has been concentrated on utilization and application of MoS_2-Sb_2O_3 solid lubricants.

At the present time, it appears that any significant advancement in solid film lubrication technology is dependent upon the development of new lubricant-additive systems or the identification of additives which, when added to MoS_2, give rise to friction and wear characteristics superior to those currently available with MoS_2-Sb_2O_3 lubricants. In light of the vast knowledge regarding solid film lubricants, it is highly unlikely that a new lubricant-additive system would be found superior to those based on MoS_2. The limited number of investigations regarding synergistic additives to MoS_2 does, however, present a possibly fruitful approach.

The current investigation is directed toward identification of the mechanism or mechanisms by which additions of Sb_2O_3 enhance the lubrication properties of MoS_2 solid film lubricants. The approach being taken includes examination of possible chemical, mechanical, and mixed chemical-mechanical effects.

1.2 SUMMARY

Investigations of possible chemical reaction between MoS_2 and Sb_2O_3 were conducted using thermogravimetric analysis. Results of this study showed distinct evidence of chemical interaction. Thermograms of MoS_2/Sb_2O_3 mixtures showed significantly lower weight losses at 500°C and 600°C than that predicted for simple mixtures of the two materials. Additionally, a small weight gain was observed prior to weight loss. This was not observed in the pure materials.

The possible formation of MoS_2-Sb_2O_3 compounds showed no chemical reaction occurring at temperatures up to 540°C. X-ray diffraction data showed MoS_2 to be oxidized to MoO_3 and Sb_2O_3 to be oxidized to Sb_2O_4. Lattice constant determinations showed little solubility of Sb_2O_4 in MoO_3 or of MoO_3 in Sb_2O_4. Sb_2O_3 additions did, however, promote the sintering of MoO_3 during oxidation.

Mechanical effects of Sb_2O_3 on MoS_2 were observed in pellet test studies. Sb_2O_3 was observed to promote the pressing characteristics of the pellets. Much lower forming pressures were required for Sb_2O_3-bearing pellets. Additionally, pellets could not be formed at high pressures.

Studies of burnished pellets showed the surface of Sb_2O_3 containing pellets to be significantly different than those of pure MoS_2. Additionally, studies of the sulfur content on the burnished surface was observed to be anomolously low in Sb_2O_3/MoS_2 mixtures.

SECTION II

REVIEW OF LITERATURE

Historically, the most important solid film lubricants have been graphite and molybdenum disulfide. These materials have been used both as solid, dry film lubricants and as additives in hydrocarbon based lubricants. The literature pertaining to the mechanisms of solid lubrication, factors affecting lubrication properties and the applications of graphite and molybdenum disulfide as solid lubricants is extensive. Excellent review articles of solid lubricants have been published by Winer[1] and by Campbell[2].

Perhaps it is only appropriate that the first reports of synergistic behavior in solid lubricants concerns additions of graphite to MoS_2[3, 4]. In 1961, Devine, Lamson, and Bowen[5] used MoS_2 and graphite mixed with sodium silicate to form a bonded solid film lubricant for high speed ball bearing applications. Their investigations clearly showed a mixture of nine parts MoS_2 and one part graphite to exhibit superior lubrication characteristics when compared to 100% MoS_2. The optimum composition resulting from their work consisted of 71 weight percent MoS_2, 7 weight percent graphite, and 22 weight percent sodium silicate.

Kay[6] found a 10 weight percent addition of graphite to MoS_2 markedly improved the lubricity of MoS_2 when tests were conducted at high humidity. However, Kay also points out that the substrate metals being lubricated played a much more important role in lubricative properties of the film than did the composition of the film itself.

The first report of nonlubricant additives giving rise to a synergistic enhancement of lubricant properties was the work of Haltner and Oliver[7]. They reported the critical parameter in solid film lubrication to be the establishment of a well formed lubricant film on the substrate. An addition of ten weight percent of stannic sulfide was observed to enhance the film forming characteristics of MoS_2. Subsequent investigation of other sulfide additives revealed several other compounds to synergistically increase the load carrying capacity of their studies (Table 1).

According to the authors, the behavior of these sulfide additions was observed to resemble that of extreme pressure additives used in liquid lubricants and, as such, their behavior was attributed to a chemical effect. Chemical analysis of the tested films indicated that the effective additives were more highly concentrated in the rubbed films than in the pellets used to establish the transfer films. In addition, it was observed that the most effective additives were those of lowest thermodynamic stability.

Lancaster[8, 9], in part, disputes the findings of Haltner and Oliver[7]. In investigating the effects of substrate surface finish on MoS_2 lubricants, Lancaster concludes that in the instance of relatively rough surface finishes, the transfer film is established and bonded to the substrate primarily by mechanical means, specifically, through the penetration of the metal by the relatively hard edges of lamillar lubricants. In the course of his investigations he made 10 weight percent additions of stannic sulfide to MoS_2 and, in the case of rough surface finished substrates, found no enhancement of the film forming characteristics of the MoS_2. He does concede, however, that in the case of highly polished substrates, chemical rather than mechanical mechanisms for film formation may be more significant and as such, SnS_2 may enhance the lubricating properties of MoS_2.

In 1965, Calhoun, et al.[10] presented a summary of more than twelve years of research on solid lubricants carried out at Rock Island Arsenal. In their efforts to improve and understand solid lubricants they selected a bonded solid film lubricant consisting of 66 weight percent MoS_2, 19 weight percent synthetic graphite and 15 weight percent epoxy-phenolic resin as a "control" lubricant. Among their findings was that natural graphite, when substituted for synthetic graphite, resulted in a 47 percent improvement in wear life.

TABLE 1

SUMMARY OF SYNERGISTIC SULFIDE ADDITIVES[7]

Additive	Coefficient of Friction	Maximum Load Carried (kg)	Additive	Coefficient of Friction	Maximum Load Carried (kg)
Sb_2S_5	0.057	5.8	ZrS_2	0.061	2.3
PtS	0.056	5.5	HgS (black)	0.048	1.8
TiS_2	0.04	5.4	Tl_2S_3	0.060	1.5
HgS (red)	0.038	5.3	Cr_2S_3	0.080	1.2
Ag_2S	0.06	5.3	BaS	0.07	1.0
PbS	0.038	5.2	Tl_2S	0.082	0.9
FeS	0.063	4.9	SnS	0.07	0.9
Ti_2S_3	0.065	4.9	CaS	0.15	0.9
Cu_2S	0.064	3.9	CdS	0.14	0.8
CuS	0.059	3.6	FeS_2	0.40	0.3
Au_2S	0.065	2.8	ZnS	--	0.3
Bi_2S_3	0.045	2.9	MoS_2	--	0.3
SnS_2	0.047	2.8			

More importantly, it was found that the addition of various nonlubricant compounds to the formulation led to enhanced wear behavior. Results of these preliminary studies are shown in Table 2.

Pursuing their investigation of synergistic additives in solid lubricants, it was found that the appropriate addition of antimony trioxide to the MoS_2 bonded lubricant, with or without the graphite constituent, resulted in Falex wear life 105 percent better than the base formulation.

As a result of these studies, G. P. Murphy and F. S. Meade[11] were granted U. S. Patent 3, 223, 636 in which several resin bonded solid lubricants were claimed. The essential constituents of these lubricants were MoS_2 and Sb_2O_3. The composition of the lubricant pigments varied from 70 to 74 weight percent MoS_2 and 30 to 26 weight percent Sb_2O_3.

TABLE 2

EFFECTS OF ADDITIVES ON FALEX WEAR LIFE OF SOLID FILM LUBRICANT*

	Concentration of Additive	
	5%	20%
Additive	% Improvement	% Improvement
Ag (powdered)	3	-14
Pb (powdered)	3	11
Sn (powdered)	3	- 4
PbO	-10	24
Sb_2O_3	20	29
Bi_2O_3	22	- 1
$MnCl_2 \cdot 4H_2O$	4	58
CdI_2	5	24
$ZnSO_4$	12	36
BN	18	27

* Wear life of base formulation = 146 minutes.

Since the reports of Haltner and Oliver and of Calhoun and his colleagues, little has been published regarding identification of new synergistic additives for MoS_2 solid film lubricants. The majority of the studies since 1965 have centered on improvement of bonded solid film lubricants and on optimization of MoS_2 to Sb_2O_3 ratios and lubricant binder ratios.

Resin bonded lubricants have received the most attention with efforts directed toward increasing both wear life of the lubricant and the high temperature stability of the resin. Since the use of epoxy phenolic resins by Calhoun, et al.[11], polyimide[12 - 15], polybenzothiazole (PBT)[16, 17], polybenzimidazole(PBI)[18, 19], and methyl phenyl polysiloxane[20, 21, 22] have received considerable attention as bonding resins for MoS_2-based solid film lubricants.

Two polyimide bonded solid lubricants were developed by Campbell and Hopkins[12]. The first formulation, designated MLR-1, consisted of a MoS_2 to Sb_2O_3, weight ratio of 3 to 1 and a lubricant-to-binder ratio of 78.8 to 21.2 by weight. The second formulation, designated MLR-2, consisted of a MoS_2 to Sb_2O_3 weight ratio of 1 to 1 and a lubricant to binder ratio of 2 to 1 by weight. Evaluation of these formulations was determined with both Falex and dual-rub shoe test devices. MLR-2 was observed to exhibit superior wear properties at room temperature and at temperatures up to 600°F (316°C).

Ling[13] used MLR-2 in a study of substrate surface roughness of friction and wear life. Hopkins and Campbell[14] used MLR-2 in their study of the effect of lubricant film thickness on wear life.

In 1969, Hopkins and Campbell[15] reported the results of evaluations of wear life tests conducted on numerous bonded solid film lubricants. Tests were conducted using the Falex tester, pellet tester, and a journal-bearing test apparatus. Among the compositions tested were several MoS_2-graphite mixtures, a MoS_2-Sb_2O_3-epoxy/phenolic mixture and MLR-2. Results of their evaluation showed no clear indication of superior friction and wear properties for the MoS_2-Sb_2O_3 based lubricants. The MLR-2 formulation was superior only in the case of the wear life test conducted on the pellet tester but this most likely is a result of the polyimide resin binder rather than of the MoS_2-Sb_2O_3 lubricant. Friction test data indicate slightly lower friction coefficients at a temperature of 400°F (204°C).

McConnell and Benzing[16] and McConnell[17] reported the use of polybenzothiazoles as binders for high temperature solid lubricant applications. Lubricants evaluated in their work included MoS_2, WS_2, TaS_2, and graphite. Lubricant additives included ZnO, TiO_2, $ZnSO_4$, $MnCl_2$, LiO_2, and Sb_2O_3. MoS_2 was clearly shown to be the superior lubricant pigment in single lubricant screening tests conducted on the Falex tester. A MoS_2 to binder ratio of 3 to 1 by weight was shown to be optimum. Interestingly, the addition of Sb_2O_3, ZnO, graphite, TiO_2, $AnSO_4$, $MnCl_2$, and LiO to the base MoS_2 lubricant resulted in reduced wear life. This is contrary to past reports regarding the synergistic enhancement of wear life resulting from additions of Sb_2O_3 and graphite.

Results from the preliminary screening of mixed lubricants indicated the best formulation to be 75 weight percent MoS_2 and 25 weight percent Sb_2O_3 and a lubricant to binder ratio of 3 to 1 by weight. It was also found that a mixture of 2 parts MoS_2 and 1 part ZnO mixed 3 to 1 with the polybenzothiazole yield excellent wear life characteristics.

Hubbell and McConnell[18] reported the use of polybenzimidazole as a high temperature solid lubricant binder. The lubricant used consisted of 55 weight percent MoS_2 and 45 weight percent Sb_2O_3. Wear-life studies were conducted to determine the optimum lubricant to binder ratio. At room temperature it was found that formulations containing 23 weight percent and 45 weight percent yield equal performance. When tested at elevated temperatures, the 23 weight percent polybenzimidazole was clearly the optimum formulation. U.S. Patent 3,721,625 was granted to McConnell, Lavik and Campbell[19] for the polybenzimidazole bonded solid lubricants.

Benzing, McConnell, and Clow[20], Benzing and McConnell[21], and Benzing, Hopkins and Petronio[22] reported on extensive research in developing a polysiloxane, methyl-phenyl-polysiloxane, as a potential room temperature curing binder for potential application in bonded solid film lubricants. The results of lubricant formulation studies[20] showed lubricants containing MoS_2 to Sb_2O_3 ratios of 1 to 1 or 3 to 2 were the preferred lubricant pigment formulations and that lubricant to binder volume ratios between 60 to 40 and 85 to 15 were preferred. One composition, designated AFSL-41, containing a MoS_2 to Sb_2O_3 weight ratio of 3 to 2 and a lubricant to binder volume ratio of 70 to 30 was found to exhibit the best overall properties. Bi_2O_3 and ZnO were also observed to result in increased wear-life performance.

The curing process was also shown to seriously affect the wear-life of MoS_2-Sb_2O_3 lubricants. All formulations containing Sb_2O_3 suffered from elevated temperature (480°F, 249°C) curing processes. The amount of degradation appeared to vary directly with the amount of Sb_2O_3 present in the formula.

Statistical evaluation of data gathered using the Falex tester, LFW-1 and journal-bearing tester was conducted using step wise regression techniques[21]. Results of these evaluations showed the preferred MoS_2 to Sb_2O_3 was 2 to 1 and that the preferred lubricant to binder ratio was 1 to 1.

Fehrenbacher, McConnell, Pellerin, and Mecklenburg[23] reported the use of a MoS_2-Sb_2O_3 mixture in producing sputter deposited solid film lubricants. Results of their studies clearly showed antimony trioxide to behave synergistically with MoS_2 in solid film lubricants.

In 1973, Lavik, Hubbell, and McConnell[24] made a unique contribution to the understanding of synergism in solid film lubrication. They hypothesized that in the process of establishing a lubricating transfer film, a low melting oxide eutectic formed from MoO_3, Sb_2O_3 and/or substrate metal oxides. The formation of such a eutectic was envisioned to act as a binder in the solid lubricant and to promote the bond between the lubricant film and the substrate. Although no direct evidence for the formation of such an oxide eutectic is presented, the role of oxygen in the formation of transfer films is implied from friction measurements made with a pellet tester.

Subsequent tests[24] using bonded solid film lubricants confirmed the synergistic behavior of Sb_2O_3-MoS_2 mixtures and indicated that Sb_2S_3 also results in enhanced lubrication characteristics though not to the extent as is observed with Sb_2O_3 additions.

2.1 SUMMARY

In summary, available literature indicated synergistic behavior in MoS_2 based solid lubricants is observed with additions of graphite, several inorganic sulfides, ZnO, Bi_2O_3 and Sb_2O_3. Antimony trioxide appears to exhibit the greatest synergistic effects followed by Sb_2S_3, Bi_2O_3, and ZnO. Lubricant to additive ratios for optimum performance vary from 1 to 1 to 3 to 1 depending on other variables such as the resins used in forming resin bonded solid film lubricants.

The vast majority of the investigations reporting enhanced lubricant performance through the use of nonlubricating additives are oriented toward the engineering of a bonded lubricant rather than the understanding of the mechanism or mechanisms responsible for the improved lubricating properties of mixed lubricant systems. Only one theory--oxide interaction--has been advanced in attempting to identify synergistic phenomena in solid film lubricants.

SECTION III

PROJECT ACTIVITY

Enhancement of the lubrication and wear properties of MoS_2 solid film lubricants resulting from additions of nonlubricant phases such as Sb_2O_3, Sb_2S_3, Bi_2O_3, and ZnO may involve both chemical and mechanical effects. Any attempt to identify the mechanism(s) resulting in the observed enhancement must assess the consequences of these additives upon a number of fundamental phenomena. Purely chemical interactions between the lubricant, the additive, the substrate, and environmental species must be investigated. Likewise, purely mechanical effects such as particle packing, film shear strength, etc., may be implicated. Most important, however, is the assessment of mixed mechanical-chemical or tribochemical effects such as formation of a lubricant-additive reaction product of intrinsically lower coefficient of friction, chemisorption of a surface film on the additive resulting in reduced shear strength in the composite film, etc. These phenomena are often subtle or intermediary and as such are most difficult to isolate experimentally. Nonetheless, this study must consider them as critical to the enhancement mechanism.

The general approach used in this study of synergistic additives has been to first search for gross alterations in the chemical and mechanical behavior of MoS_2 brought on by the addition of controlled amounts of additive. Antimony trioxide was selected as the additive since it is reported to give the most pronounced effect. Mixtures containing 25 volume percent Sb_2O_3 and 45 volume percent Sb_2O_3 were selected for study and to be compared to pure MoS_2. Results obtained from these preliminary investigations are then used to identify definitive experiments for isolation and identification of mechanico-chemical effects. Owing to the complexity of lubrication and wear phenomena each experiment has been designed to answer a specific question. In this way, the role played by Sb_2O_3 additions may be identified by alternative hypotheses.

3.1 OXIDATION

Are the oxidation kinetics of MoS_2 affected by the presence of Sb_2O_3?

Both oxygen and water vapor have been shown to exhibit a pronounced effect of the coefficient of friction and the wear life of MoS_2 dry lubricants [25] as well as on the establishing of good transfer films [24]. Lavik discussing the work of Gansheimer [25] points out that in the Falex tester, energy input at the frictional interfaces may be as high as 120 watts for a coefficient of friction of 0.10, a 1000 pound jaw load and a surface speed of 19 ft. per minute. This should be sufficient energy to promote some chemical reaction in the lubricant or between lubricant and substrate or lubricant and atmosphere.

One might propose, therefore, that the lubrication and wear properties of Sb_2O_3/MoS_2 mixtures are enhanced by some modification of the oxidation kinetics. For this reason, thermogravimetric analysis of pure MoS_2, pure Sb_2O_3, 25 weight percent Sb_2O_3-75 weight percent MoS_2, and 45 weight percent Sb_2O_3-55 weight percent MoS_2 were carried out. Each powder was analyzed in both dry and wet air. Wet air was produced by bubbling the dry inlet air through a water bath at room temperature. All analyses were performed in duplicate. The resulting thermograms are presented in Figures 1 through 6.

Figure 1 is the TGA thermogram for MoS_2. The initial weight loss reaction occurs at 300°C and is completed by 490°C. This corresponds to the oxidation of MoS_2 to MoO_3. The observed weight loss - 10.95% - compares favorably with the expected weight loss of 10.08%. The second reaction beginning at 655°C corresponds to the distillation of MoO_3 from the TGA sample container. Melting of residual MoO_3 was observed at approximately 795°C.

Figure 2 shows the thermogram for pure Sb_2O_3. Oxidation of Sb_2O_3 to Sb_2O_4 or to Sb_3O_5 would require weight grains of 5.49% and 10.98%, respectively. Instead, a weight loss reaction is observed starting at

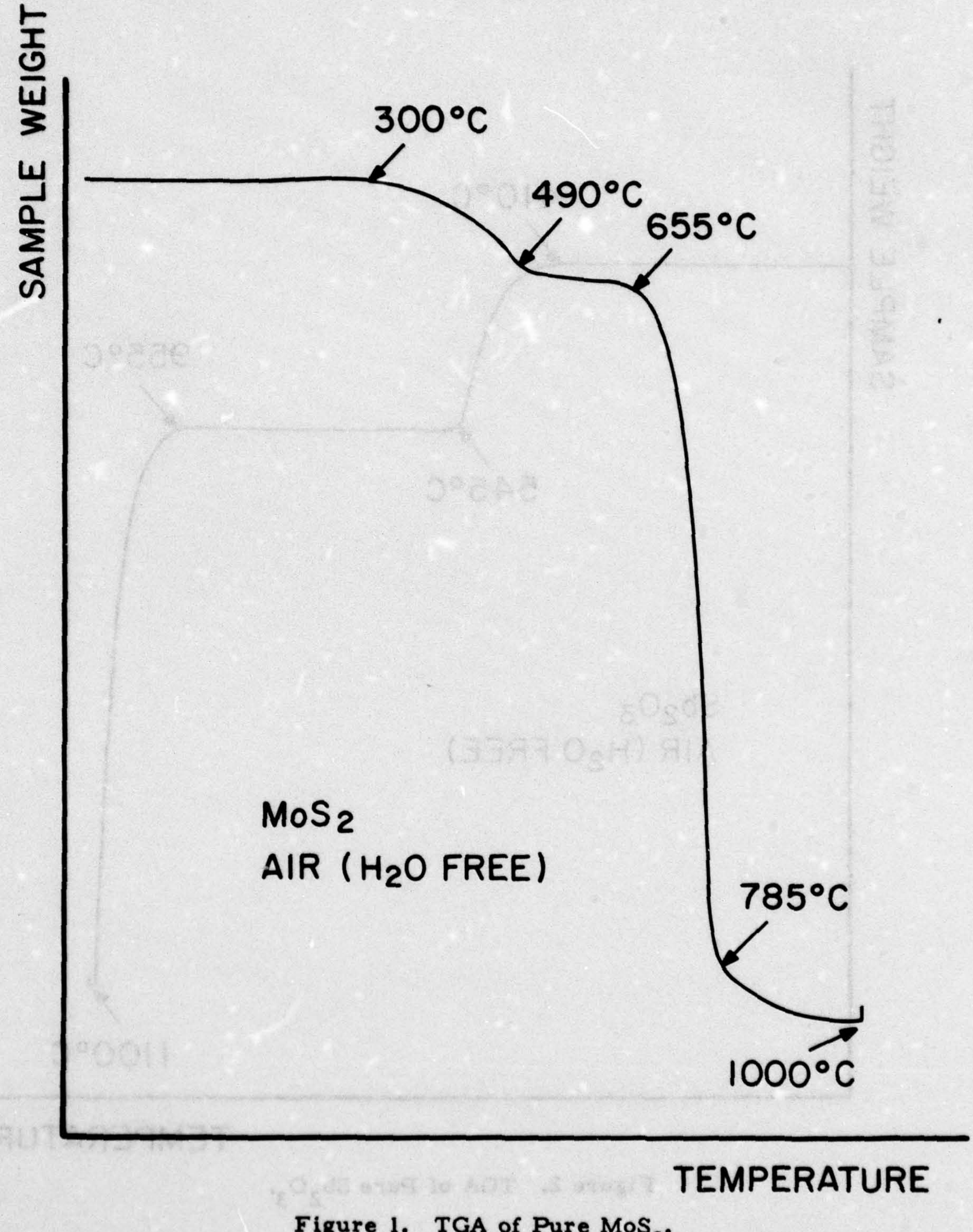


Figure 1. TGA of Pure MoS_2.

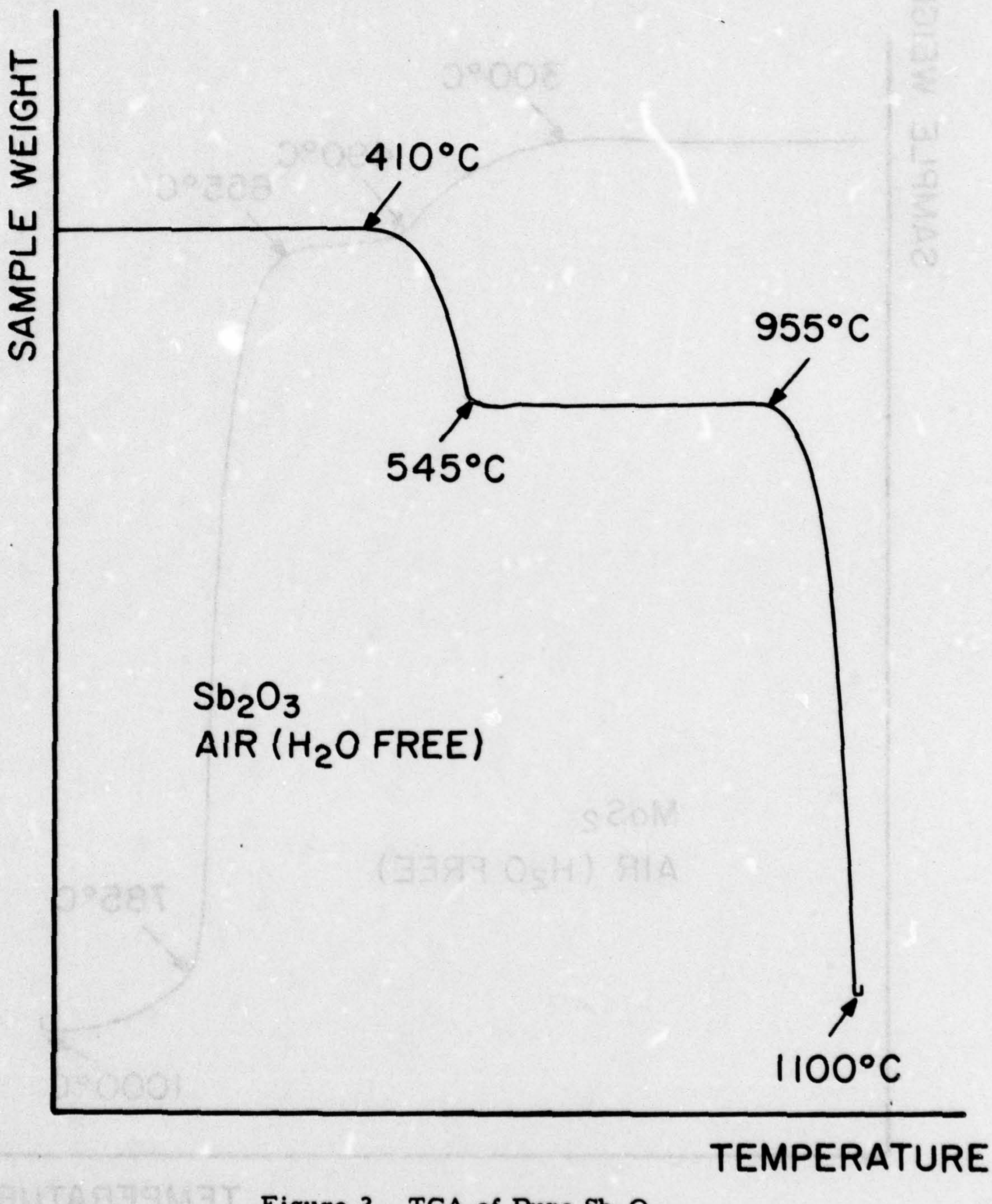


Figure 2. TGA of Pure Sb_2O_3.

approximately 410°C. The total weight loss observed for this reaction is approximately 22%. Reduction of or decomposition of Sb_2O_3 to antimony metal is ruled out because such a reaction would result in a weight loss of only 16.5%. X-ray diffraction studies of Sb_2O_3 heated to 600°C for twenty-four hours showed the Sb_2O_3 to be oxidized to Sb_2O_4. Thus, the end product of the reaction beginning at 410°C is an oxidation product of Sb_2O_3.

In an effort to explain the 22% weight loss which accompanies the oxidation of Sb_2O_3 to Sb_2O_4, several mechanisms were proposed. The thermal properties of the various antimony oxides show that Sb_2O_3 melts at 655°C, Sb_2O_4 decomposes at 930°C, and Sb_2O_5 decomposes at 400°C. If one considers a reaction in which Sb_2O_3 forms an unstable intermediate compound which immediately decomposes, causing antimony to be lost from the sample, the weight loss observed may be accounted for. One possible reaction might be:

$$4Sb_2O_3 + 5/2O_2 \rightarrow 3Sb_2O_4 + Sb_2O_5$$
$$Sb_2O_5 \rightarrow 2Sb\uparrow + 5/2O_2\uparrow.$$

The calculated weight loss for such a reaction is 20.9% which is in fair agreement with the observed 21.7%. Additionally, the decomposition temperature of Sb_3O_5 (400°C) is in good agreement with the observed 410°C starting temperature for the reaction. Further TGA work and possibly isothermal weight loss studies will be required to completely understand this reaction.

A second weight loss reaction in the Sb_2O_3 thermogram was observed to start at approximately 955°C and corresponds to the decomposition of Sb_2O_4.

Figures 3 and 4 show the thermograms for the 25% Sb_2O_3-75% MoS_2 mixture obtained in dry and in room temperature saturated air. Comparison of the two thermograms indicates the presence of water vapor results in little change in the thermal behavior of the mixture. Significant changes are

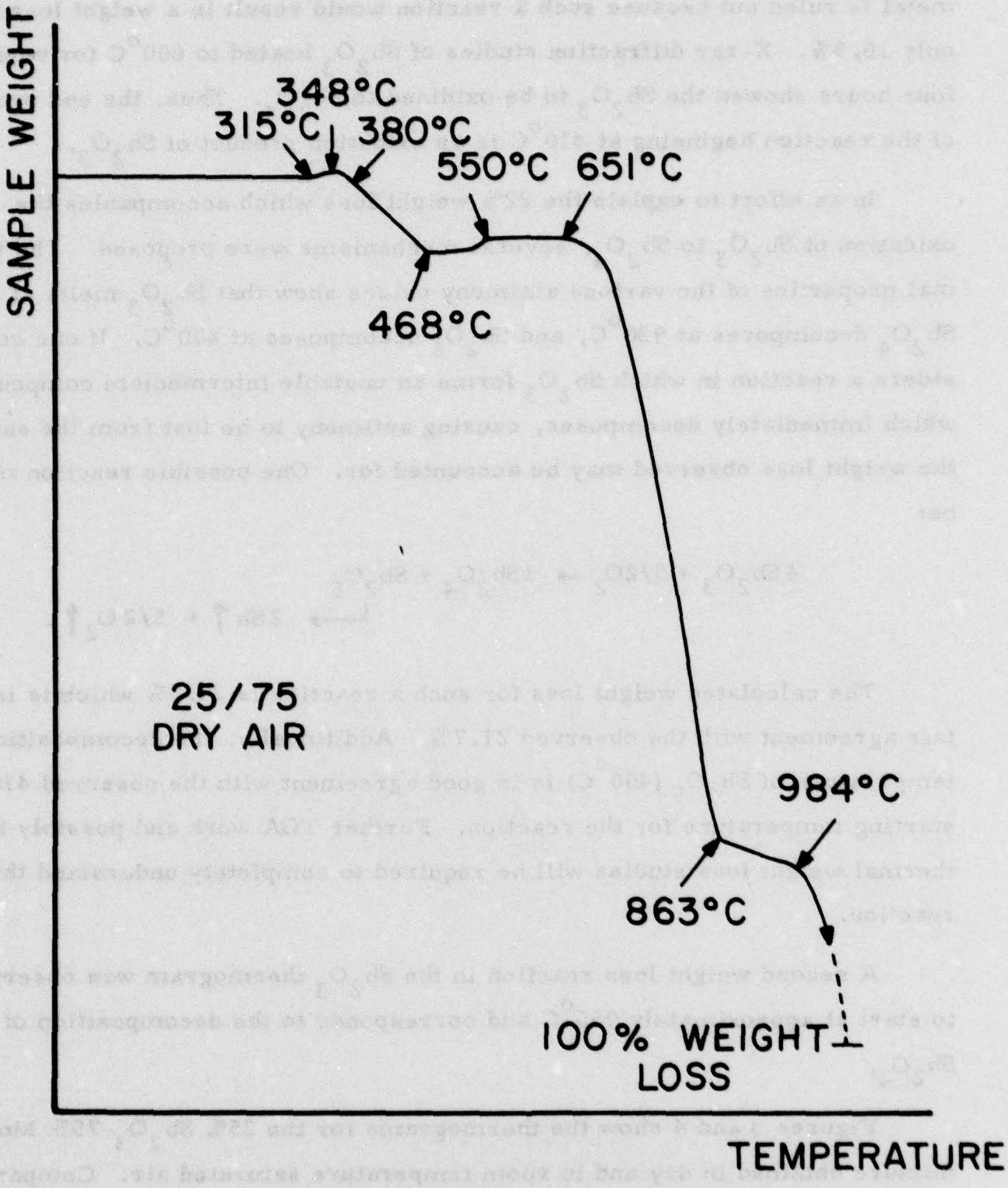


Figure 3. TGA of 25 Wt. % Sb_2O_3-75 Wt. % MoS_2 in Dry Air.

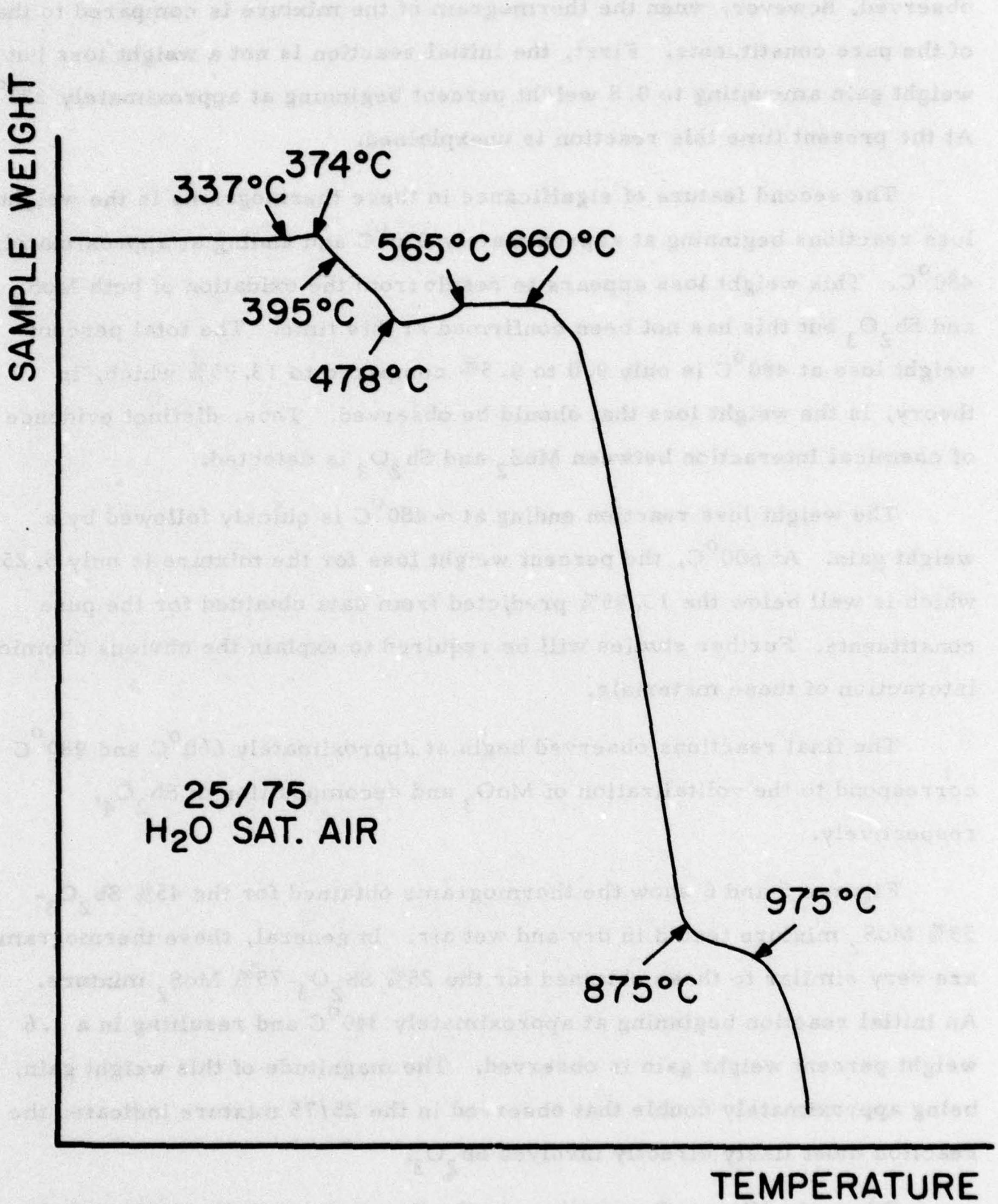


Figure 4. TGA of 25 Wt. % Sb_2O_3-75 Wt. % MoS_2 in Saturated Air.

observed, however, when the thermogram of the mixture is compared to that of the pure constituents. First, the initial reaction is not a weight loss but weight gain amounting to 0.8 weight percent beginning at approximately 320°C. At the present time this reaction is unexplained.

The second feature of significance in these thermograms is the weight loss reactions beginning at approximately 370°C and ending at approximately 480°C. This weight loss appears to result from the oxidation of both MoS_2 and Sb_2O_3 but this has not been confirmed at this time. The total percent weight loss at 480°C is only 9.0 to 9.5% compared to 13.95% which, in theory, is the weight loss that should be observed. Thus, distinct evidence of chemical interaction between MoS_2 and Sb_2O_3 is detected.

The weight loss reaction ending at ~480°C is quickly followed by a weight gain. At 600°C, the percent weight loss for the mixture is only 5.25% which is well below the 13.95% predicted from data obtained for the pure constituents. Further studies will be required to explain the obvious chemical interaction of these materials.

The final reactions observed begin at approximately 660°C and 980°C correspond to the volitalization of MoO_3 and decomposition of Sb_2O_4, respectively.

Figures 5 and 6 show the thermograms obtained for the 45% Sb_2O_3-55% MoS_2 mixture tested in dry and wet air. In general, these thermograms are very similar to those obtained for the 25% Sb_2O_3-75% MoS_2 mixture. An initial reaction beginning at approximately 340°C and resulting in a 1.6 weight percent weight gain is observed. The magnitude of this weight gain, being approximately double that observed in the 25/75 mixture indicates the reaction most likely directly involves Sb_2O_3.

Other significant observations in the thermogram indicate the weight loss at 500°C and 600°C are far less than would be expected if no reaction occurred between Sb_2O_3 and MoS_2. Based on weight losses observed in pure

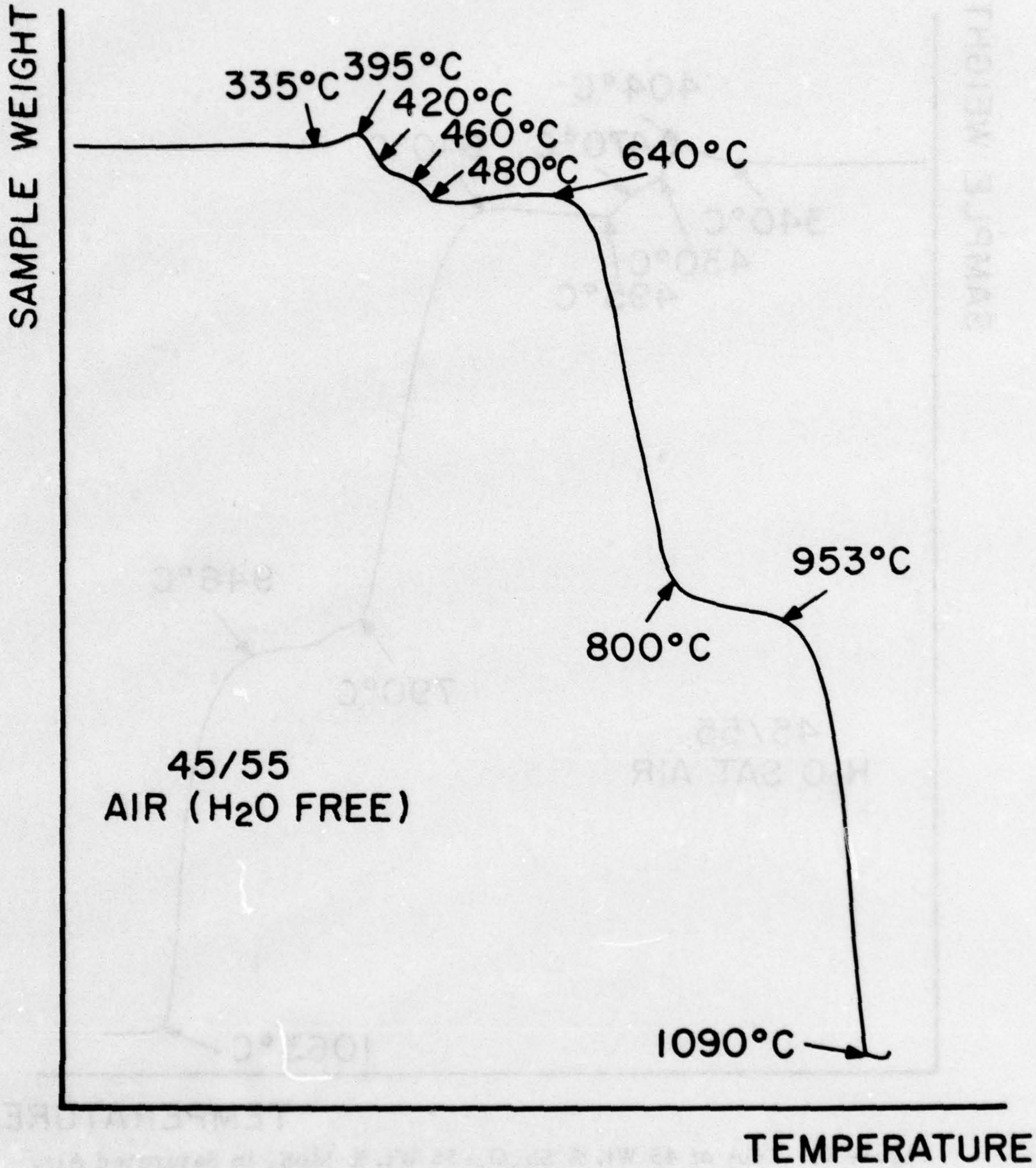


Figure 5. TGA of 45 Wt. % Sb_2O_3-55 Wt. % MoS_2 in Dry Air.

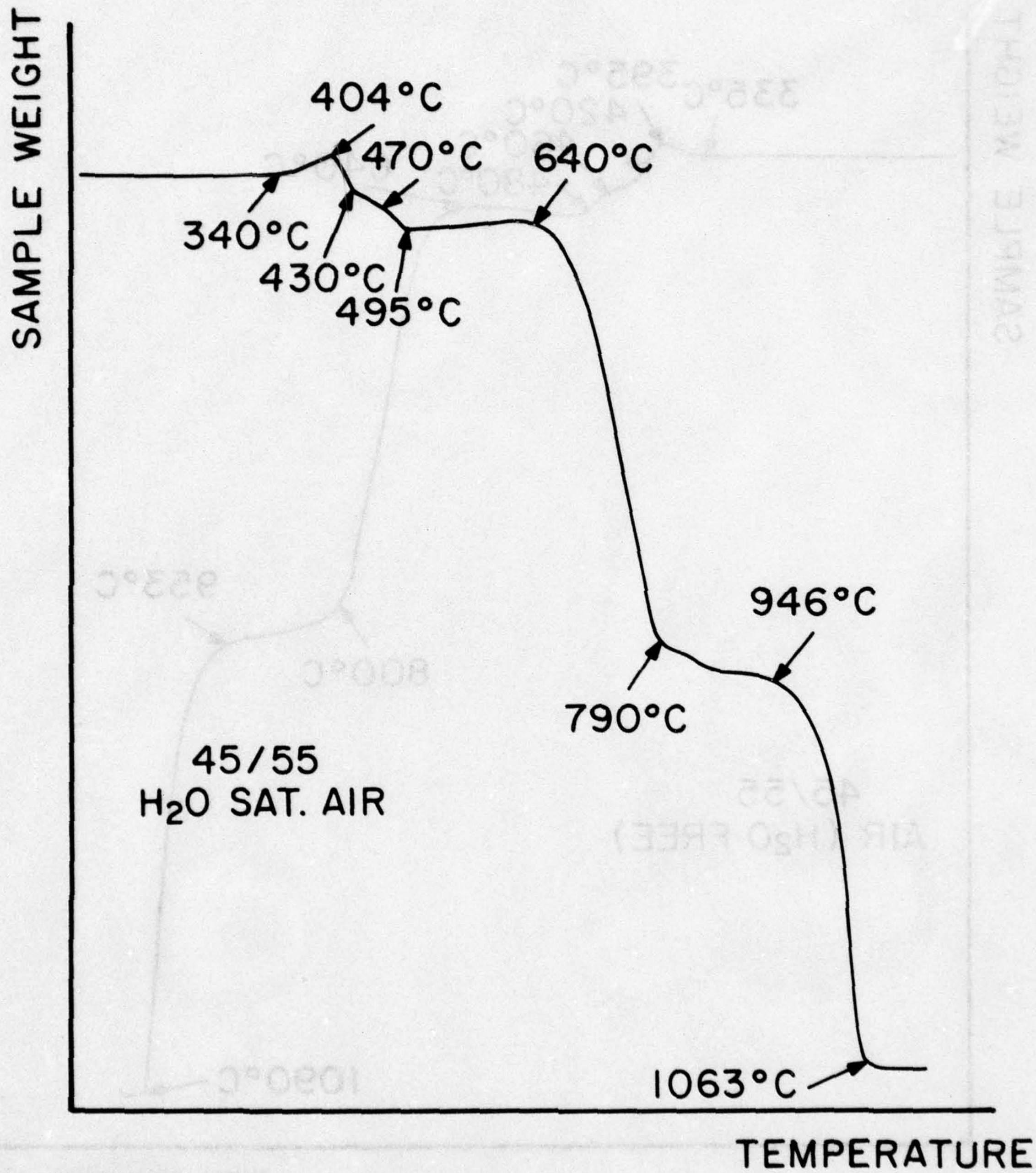


Figure 6. TGA of 45 Wt. % Sb_2O_3-55 Wt. % MoS_2 in Saturated Air.

MoS_2 and pure Sb_2O_3, a 15.83% weight loss would be expected at 500 and 600°C. Instead, weight losses of only 6.5% and 5.65% were observed at those temperatures. Further study is required to explain these reactions.

The final reactions in the thermogram correspond to volitalization of MoO_3 and decomposition of Sb_2O_4 as was observed in previous studies.

In summary, TGA data indicates Sb_2O_3 interacts with MoS_2 in the temperature range 300-600°C resulting in a pronounced alteration of the oxidation behavior of MoS_2. Further investigations, including TGA in oxygen free atmosphere and possibly sulfur bearing atmospheres (SO_2 or H_2S) as well as isothermal weight loss studies are required if the exact nature of the observed interaction MoS_2 and Sb_2O_3 is to be fully understood.

3.2 NEW PHASE FORMATION

What are the reaction products formed upon heating MoS_2/Sb_2O_3 mixture?

Thermogravimetric analysis of Sb_2O_3/MoS_2 mixtures indicated interaction of the two powders resulting in weight losses far less than predicted from pure constituent data. In order to understand this interaction, a preliminary study was performed in which the 25%/75% mixture, the 45%/55% mixture, and pure MoS_2 were lightly compacted into small pellets and heated in air to 540°C (1004°F) for 16 hours. The role of water vapor in these studies was ignored based on earlier TGA studies. The temperature of 540°C was selected since it represents complete oxidation of MoS_2 to MoO_3 as shown by TGA. Possible reaction couples anticipated in this investigation included MoS_2 with Sb_2O_3, MoO_3 with Sb_2O_3, and MoO_3 with Sb_2O_4. Following heating, the samples were examined with the scanning electron microscope (SEM) and by x-ray diffraction (XRD).

In preparation of the reacted powder mixtures for analysis, significant differences were observed between the specimens. First, the shrinkage of the powder compacts increased with increasing Sb_2O_3 content. Second, while the pure MoS_2 compact easily crumbled following heating, the compacts containing Sb_2O_3 were found to be hard, dense pellets typical of sintered materials.

X-ray diffraction studies of the oxidized powders indicated that in every pellet, MoS_2 was oxidized to MoO_3 and Sb_2O_3 was oxidized to Sb_2O_4. No MoO_3-Sb_2O_4 compounds were found.

The XRD data indicate no compound formation between MoS_2 and Sb_2O_3 when mixtures were heated in air. Additionally, computer refined lattice parameters indicated little or no solid solubility between the MoO_3 and Sb_2O_4. These data coupled with the qualitative observations regarding shrinkage and densification of the oxides strongly indicate the likelihood of the formation of a MoO_3-Sb_2O_4 eutectic. Further sintering studies and differential thermal analysis studies will be required to confirm this possibility.

Scanning electron microscope examination confirmed the macroscopic observations made regarding the sintering of the oxide powders. The pure MoS_2 pellet oxidized to MoO_3 was still particulate in nature. Those pellets containing Sb_2O_3 contained numerous large, dense grains.

In summary, two conclusions may be drawn from this experiment. First, the formation of an antimony-molybdenum compound is not observed upon heating the sulfide-oxide mixture. The MoS_2 oxidizes to MoO_3 as was anticipated while Sb_2O_3 is oxidized to Sb_2O_4. Secondly, densification or sintering was observed to occur during heating MoS_2/Sb_2O_3 mixtures and was not observed when heating pure MoS_2. The exact nature of the MoO_3-Sb_2O_4 interaction is not fully understood at this time but merits further study due to its potential significance in MoS_2-Sb_2O_3 synergism.

In order to further understand the effect of Sb_2O_3 on the oxidation behavior of MoS_2, a quantitative investigation was begun. Pellets of pure MoS_2, 75% MoS_2 + 25% Sb_2O_3 and 55% MoS_2 + 45% Sb_2O_3 were pressed at 172 MPa (25,000 psi), at 345 MPa (50,000 psi), and at 690 MPa (100,000 psi).

The height, diameter, and weight of each pellet were measured. The pellets were then heated at various temperatures between 300°C and 600°C to study the oxidation reactions occurring in that temperature range. Following oxidation, the pellets were again measured and weighed, and to determine the amount of oxidation of MoS_2 and/or Sb_2O_3, quantitative x-ray diffraction studies were performed.

Table 3 summarizes the changes in volume, mass, and density observed in the 300°C oxidation study. The significant results in these data are that those pellets containing no Sb_2O_3 consistently lost weight where as those containing the Sb_2O_3 additions all gained weight. Additionally, the pellets containing 45% Sb_2O_3 additions underwent essentially no change in density. This indicates that Sb_2O_3 acts to reduce the oxidation of MoS_2 to MoO_3.

In order to confirm the apparent lack of oxidation of Sb_2O_3 containing pellets, quantitative x-ray diffraction analysis was performed on the oxidized pellets.

From x-ray diffraction theory, we know that the intensity of the diffracted x-rays from a crystallographic plan (hkl) is a function of the volume of material being irradiated. Specifically,

$$I_{hkl} = \frac{I_o C j F_{hkl}^2 V(L-p)}{\mu}$$

where I_{hkl} is the intensity of the diffracted beam, I_o is the intensity of the incident beam, C is an experimental constant, j is the multiplicity of planes (hkl), F_{hkl}^2 is the structure factor, V is the volume scattering x-rays, (L-p) is the Lorentz-polarization factor, and μ is the linear absorption coefficient.

For a mixture of phases, it can be shown that, if the linear absorption coefficients of the phases in the mixture are equal, a linear relationship between the intensity of any diffraction maximum, I_{hkl}, and the volume fraction of that phase may be established. Thus, by determining the intensity, I_{hkl}, the volume fraction of the phase present in the sample may be determined.

TABLE 3

PHYSICAL PROPERTIES OF OXIDIZED PELLETS

Composition	Forming Pressure	Volume As Pressed	Mass As Pressed	Volume 300°C - 24 hrs.	Mass 300°C - 24 hrs.	Volume 300°C - 96 hrs.	Mass 300°C - 96 hrs.
100% MoS_2	172 MPa	0.201	0.6325	0.209	0.6286		
	345 MPa	0.172	0.5940			--*	0.5758
		0.162	0.5597			--*	0.5478
	690 MPa	0.148	0.5535	0.155	0.5590		
75% MoS_2 -	172 MPa	0.180	0.5975	0.181	0.6028		
	345 MPa	0.169	0.6144			0.185	0.6363
25% Sb_3O_3		0.164	0.5913			0.172	0.6066
	690 MPa	0.144	0.5674	0.144	0.5697		
55% MoS_2 -	172 MPa	0.159	0.6035	0.160	0.6096		
	345 MPa	0.156	0.5851			0.160	0.6024
45% Sb_2O_3		0.148	0.5480			0.151	0.5615
	690 MPa	0.148	0.5968	0.149	0.5996		

Composition	Forming Pressure	Density As Pressed	Density 300°C - 24 hrs.	Density 300°C - 96 hrs.
100% MoS_2	172 MPa	3.147	3.008	
	345 MPa	3.453		--*
		3.455		--*
	690 MPa	3.740	3.606	
75% MoS_2 -	172 MPa	3.319	3.330	
	345 MPa	3.636		3.239
25% Sb_2O_3		3.605		3.527
	690 MPa	3.940	3.956	
55% MoS_2 -	172 MPa	3.796	3.810	
	345 MPa	3.751		3.765
45% Sb_2O_3		3.709		3.719
	690 MPa	4.032	4.051	

* Specimen fragmented during oxidation.

If, however, the linear absorption coefficients of the phases in the mixture are not equal, the relationship between volume fraction and intensity will not be linear. In these instances, which is the most common case, it is necessary to use the internal-standard technique developed by L. E. Alexander and H. P. Klug. Using this method, a series of mixtures of a standard material and this phase of interest are prepared, and values of I_{hkl} for the standard and $I_{h'k'l'}$ for the phase of interest are determined. The ratio of the two intensities is then proportional to the volume fraction of the phase being studied.

In this investigation, mixtures of MoS_2 and MoO_3, 75% (MoS_2 + MoO_3) + 25% Sb_2O_3 and 55% (MoS_2 + MoO_3) + 45% Sb_2O_3 were prepared. Following thorough mixing, the x-ray diffraction pattern for each mixture was determined. Specific diffraction maxima were selected and the ratios,

$$\frac{I_{004}^{MoS_2}}{I_{400}^{Sb_2O_3}}, \quad \frac{I_{040}^{MoO_3}}{I_{400}^{Sb_2O_3}}, \quad \text{and} \quad \frac{I_{040}^{MoO_3}}{I_{004}^{MoS_2}}$$

were calculated and plotted against the volume fraction of MoO_3. Unfortunately, with the exception of the $I_{040}^{MoO_3} / I_{004}^{MoS_2}$ ratio, the calibration curves were neither linear nor systematic. This was attributed to the extreme preferred orientation problems inherent in working with layer structures such as MoS_2 or with highly acicular crystals such as MoO_3. Numerous unsuccessful attempts were made to circumvent the orientation problems.

X-ray diffraction patterns obtained for the MoS_2 and MoS_2/Sb_2O_3 pellets oxidized at 300°C, 24 hours and 96 hours showed that in all pellets containing Sb_2O_3, no MoO_3 was detectable. From calibration curves, the approximate weight fraction of MoO_3 present in the oxidized MoS_2 was 7 percent for the pellet pressed at 345 MPa. The apparent variation in amount of oxide formed with forming pressure is not understood.

X-ray studies of the pellets oxidized at 600°C show that all MoS_2 was oxidized to MoO_3 and that Sb_2O_3, if present, was oxidized to Sb_2O_4.

In summary, it has been shown that the presence of Sb_2O_3 inhibits the oxidation of MoS_2. This finding indicates a potential explanation for the observed increase in the wear life of MoS_2 solid film lubricants. The oxidation of MoS_2 at the lubricant-substrate interface will lead to premature failure of the lubricant film. Either prevention or retardation of this oxidation would extend the life of the film. Further investigation of the oxidation process in MoS_2-Sb_2O_3 mixtures is suggested in order to further delineate the mechanism by which Sb_2O_3 inhibits the oxidation of MoS_2.

3.3 PELLET TESTER

What surface chemical and morphological changes occur upon burnishing MoS_2/Sb_2O_3 mixtures?

Results of the oxidation studies of MoS_2 and MoS_2-Sb_2O_3 mixtures showed that the presence of Sb_2O_3 causes densification and sintering. In order to attach any significance to these observations, the possibility of densification and sintering occurring during mechanical burnishing should be investigated. In addition, general changes in surface chemistry and composition during mechanical burnishing of powder mixtures must be characterized.

An instrumented PT-300 pellet tester was set up and calibrated for a 387 gm loading condition. Pure MoS_2 and the mixtures 25% Sb_2O_3/75% MoS_2 and 45% Sb_2O_3/55% MoS_2 were compacted into pellets at 172 MPa (25,000 psi). The resulting pellets were lightly burnished by rubbing on a silk polishing cloth so as to produce an identical and reproducible starting surface finish on all pellets. A 440C stainless steel wear plate was utilized; the surface cleaned for each test by polishing against 600 grit silicon carbide polishing paper and rinsing in acetone. Each set of pellets were run for one hour at 500 rpm in laboratory air. The relative humidity was approximately 45% during testing.

It is emphasized that the intent of this experiment was not to measure friction coefficients, although they were monitored in order to insure that standard conditions were being met. Rather, the intent is to produce a method for controlled preparation of transfer films and rubbing surfaces. A major requirement is to yield surfaces or films which are suitable for analytical study.

Figures 7 and 8 present SEM micrographs for the pure MoS_2 and 45% Sb_2O_3/55% MoS_2 pellet rub surfaces before and after burnishing with the pellet tester. The 100% MoS_2 surface appears as a low density, granular surface. Densified regions, although present on the burnished surface, are not generally in evidence. The 45/55 pellet by contrast, appears denser in the before burnishing condition and significantly denser in the after tested condition. This is especially evident in Figure 9.

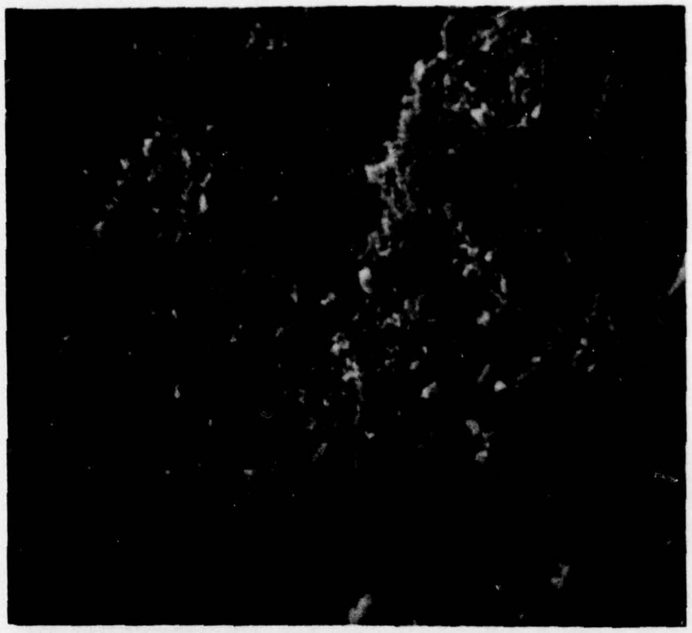
Figure 9 shows a region of the 45/55 pellet in which a distinctive dense surface film has developed. Relatively large regions of the film, separated by cracks are apparent. A small "low" density region is also present in the micrograph and is thought to result from the removal of the dense surface film during testing or during preparation of the specimen for examination.

The chemical make-up of the pellet rub surfaces was determined by Auger electron spectroscopy (AES). Samples of each composition were examined before and after burnishing. In addition, AES spectra were obtained as a function of depth into the pellet from the rubbed surface.

a) As Pressed, 100X

b) 1 hr., 550 rpm, 100X

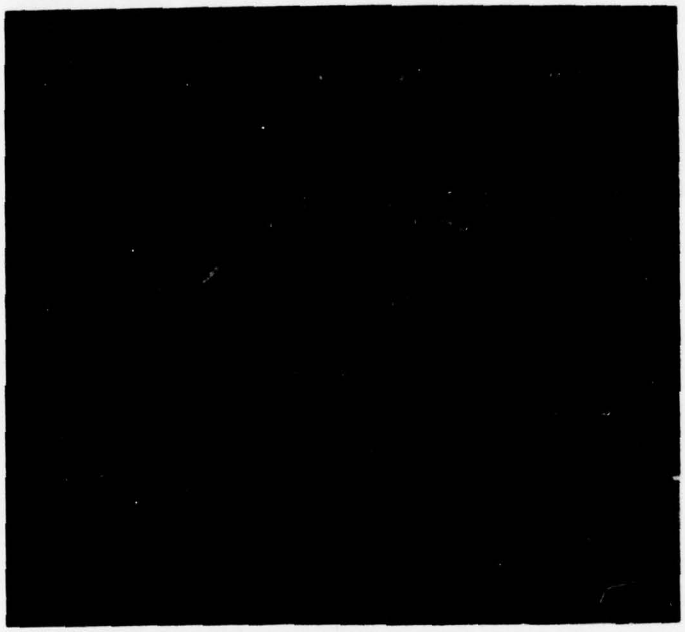
Figure 7. Wear Surface of Pure MoS_2 Pellet.



a) As Pressed, 300X

b) 1 hr., 550 rpm, 300X

Figure 8. Wear Surface of 45 Wt. % Sb_2O_3-55 Wt. % MoS_2 Pellet.



100X

Figure 9. Wear Surface of 45 Wt. % Sb_2O_3-55 Wt. % MoS_2 Pellet.

Analysis of Auger data was accomplished by measuring the height of the principal sulfur, molybdenum, antimony, and oxygen Auger peaks. It was felt that the ratio of the sulfur peak height to the molybdenum peak height offered an excellent reference for comparison between samples since this ratio should, theoretically, be independent of the presence of Sb_2O_3 (i.e. assuming a mechanical mixture). Hence, any chemical effects induced by the presence of Sb_2O_3 may be borne by differences in the S/Mo ratio. Results are shown in Figure 10.

As-pressed pellets gave a constant S/Mo ratio of approximately 4.2. After sputtering the surface of the as-pressed pellets to an approximate depth of 50 nm (500 Å), the S/Mo ratio decreased to about 2.3. It is emphasized that the observed S/Mo ratios were independent of Sb_2O_3 content in the as-pressed pellets. Pellets burnished on the pellet tester gave S/Mo ratios of 7.8 for pure MoS_2, 6.9 for the 25/75 mixture, and 5.7 for the 45/55 mixture. After sputtering approximately 50 nm, the S/Mo ratio for the burnished pellets approached the same value as that for the as-pressed pellets, 2.3.

Initial interpretation of these results suggested, (1) burnishing resulted in concentration of sulfur at the rubbed surface, and (2) Sb_2O_3 additions inhibited this sulfur concentration. Under closer examination, however, it was observed that the intensity of the sulfur Auger peak was constant at all sputtering depths and that the molybdenum Auger peak was observed to increase in height by a factor of 2.5 to 5 times depending on the sample being tested. This behavior is unexplained at the present time and is receiving further study.

Ancilliary to the results of the pellet tester, it was observed that the pellet composition exerted a strong influence on the pressing characteristics. For pure MoS_2 pellets, maximum strength was achieved at high compacting pressures (690 MPa, 100,000 psi). For the MoS_2 (Sb_2 (Sb_2O_3 mixtures, however, the optimum forming pressures appear to be in the range of 105 to 210 MPa (15-30,000 psi). This clearly indicates that Sb_2O_3 alters the

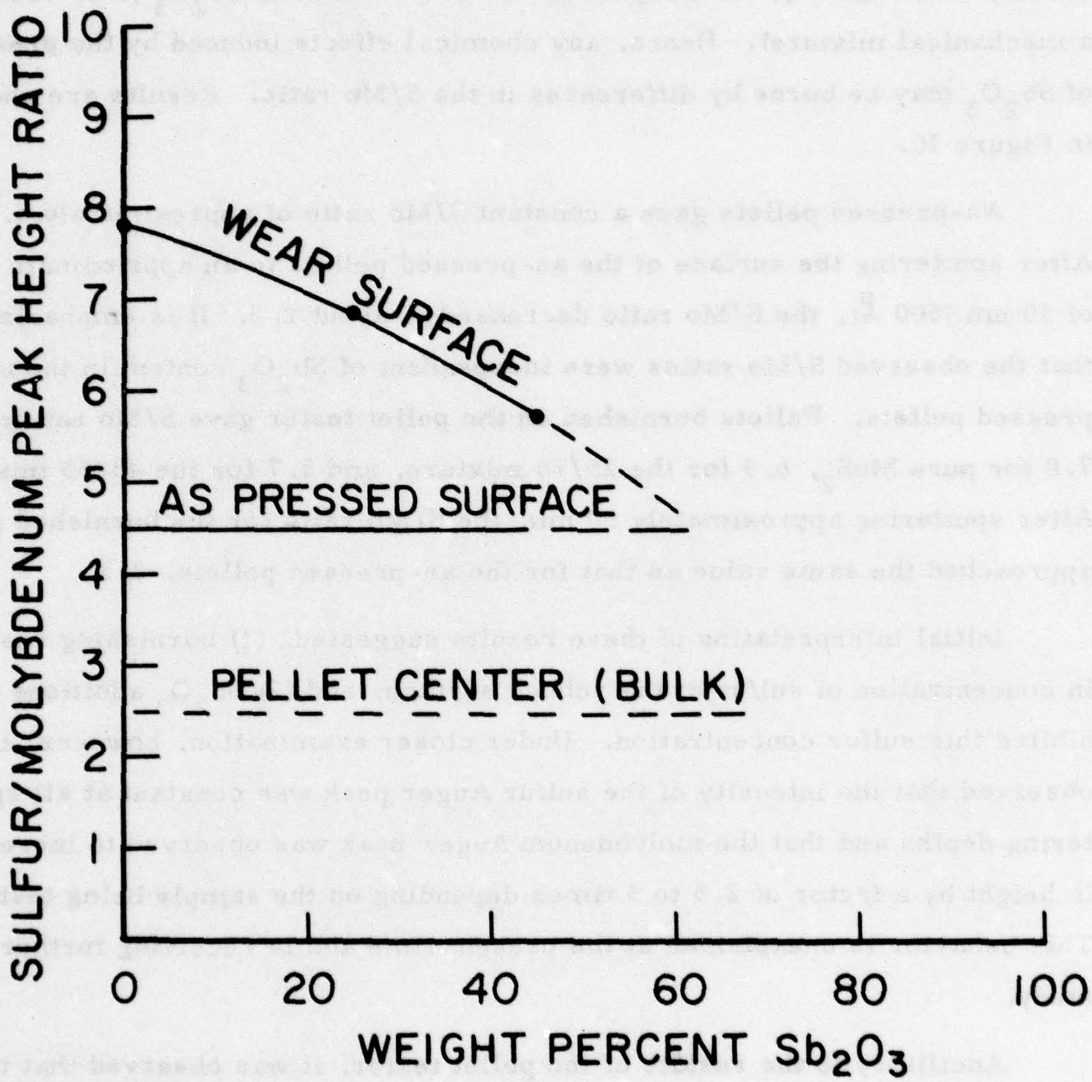


Figure 10. Sulfur/Molybdenum Auger Peak Ratio for MoS_2 and MoS_2-Sb_2O_3 Wear Surfaces.

particle packing characteristics of MoS_2. It may also suggest that Sb_2O_3 aids in particle-particle cohesion in the compact. Additionally, the powder packing characteristics may provide another means to assess the ability of the lubricant mixture to form effective transfer films during burnishing. Further study will be necessary to establish these relationships.

3.4 CONCLUSIONS AND FUTURE WORK

Results of investigations currently indicate:

1. Sb_2O_3 exhibits a pronounced effect on the oxidation behavior of MoS_2, especially in the temperature range of 300-600°C.

2. Sb_2O_3 or its oxidation product, Sb_2O_4, react with MoO_3 to form a dense, sintered product.

3. Sb_2O_3 alters the morphology of the burnished pellet surface and of the transfer film established with a pressed pellet.

4. Sb_2O_3 alters the chemistry of the burnished surface in pellets subjected to friction testing.

As indicated earlier, further clarification is required to fully understand some of the above observations.

The composition, chemistry, and morphology of the sliding interface are controlled by dynamic equilibrium conditions. Hence, differences in chemistry and morphology of the transfer films reflect the role of Sb_2O_3 additions.

Future work must be conducted to answer the following questions regarding the role of Sb_2O_3 additions in MoS_2 lubricants.

1. Does the microstructure of the transfer film--density, morphology, strength, etc.--vary significantly with Sb_2O_3 additions?

2. Does segregation of Sb_2O_3 or Sb_2O_4 occur at the sliding interface, or does Sb_2O_3 or Sb_2O_4 react with MoO_3 at the sliding interface, thereby affecting the reactivity of lubricant surface with oxygen and/or water vapor?

3. What is the role of water vapor in the Sb_2O_3 enhancement of MoS_2 lubricants?

4. What changes in surface chemistry affect the lubricant properties of the MoS_2/Sb_2O_3 film, esp. the role of sulfur?

In addition to characterization of transfer film chemistry and microstructure, a study should be made of the density and strength of powder compacts made from Sb_2O_3/MoS_2 mixtures at different compaction pressures. This study should attempt to model the effect of load upon particle packing. Hopefully, it would also shed light upon the purely mechanical effects of Sb_2O_3 upon transfer film formation.

SECTION IV

FRICTION TEST APPARATUS

4.1 ANALYTICAL CONSIDERATIONS

In the current study of synergistic mechanisms operating in the application of solid lubricants, it is emphasized that analysis of test specimens will require the use of x-ray diffraction analysis (XRD), scanning electron microscopy (SEM), electron microprobe analysis (EMP), electron spectroscopy for chemical analysis (ESCA), and Auger electron spectroscopy (AES). The analytical techniques impose severe restrictions of the method by which test specimens may be prepared, for the specimens must be flat, small in area (10 sq. mm), and comparatively thin (2-3 mm). Analytical considerations also require that the lubricant be subjected to known stress of relatively high order (100,000 to 200,000 psi) as the wear tests are performed.

4.2 DESIGN REQUIREMENTS

Along with accommodating the analytical considerations, the following design requirements were specified:

1. Load: Infinitely variable - 0 to 200,000 psi.

2. Speed: Variable - 0 to 50 fpm.

3. Test Specimen Substrate: 52100 or 440 C stainless steel hardened to 62 Rockwell C; flat and thin; test area approximately 1" x 3".

4. Environment: The apparatus must be operable in a controlled environment such as inert gas, or specified conditions of temperature and humidity.

5. The coefficient of friction must be continuously determinable in order to terminate the test at the time when breakdown occurs, or when a specified level of friction is exceeded.

6. On shutdown, the specimen must be removable and the test area left intact for analysis.

7. Assembly and disassembly of the apparatus must be accomplished with ease by a qualified technician.

A survey of lubricant testing devices in current use such as Falex, LFW-1, dual rub shoe, etc., revealed none which would adapt to the above requirements. This determined the necessity for a new design.

4.3 DESIGN DESCRIPTION

4.3.1 Design Components and Assembly

The design requirements were incorporated in the device shown in Figure 1. A cross-sectional view is shown. The components, with accompanying figure numbers are as follows:

Item	Name	Figure
1	Support Arm	12
2	Piston (2 required)	13
3	Cylinder	14
4	Cover (2 required)	15
5	Mounting Block	16
6	Shaft	17
7	Disc	18
8	Bearing Mount	19
9	Mounting Plate	20

Referring to Figure 11, Support Arm 1 bolts to Mounting Block 5, which is bolted to Mounting Plate 9. The Cylinder Assembly consists of Cylinder 3, Piston(s) 2, and Cover(s) 4. This assembly is bolted to Support Arm 1. Bearing Mount 8 bolts to Mounting Plate 9, and Shaft 6 projects through the bearing. Disc 7 is mounted on Shaft 6 and is retained by means of a nut.

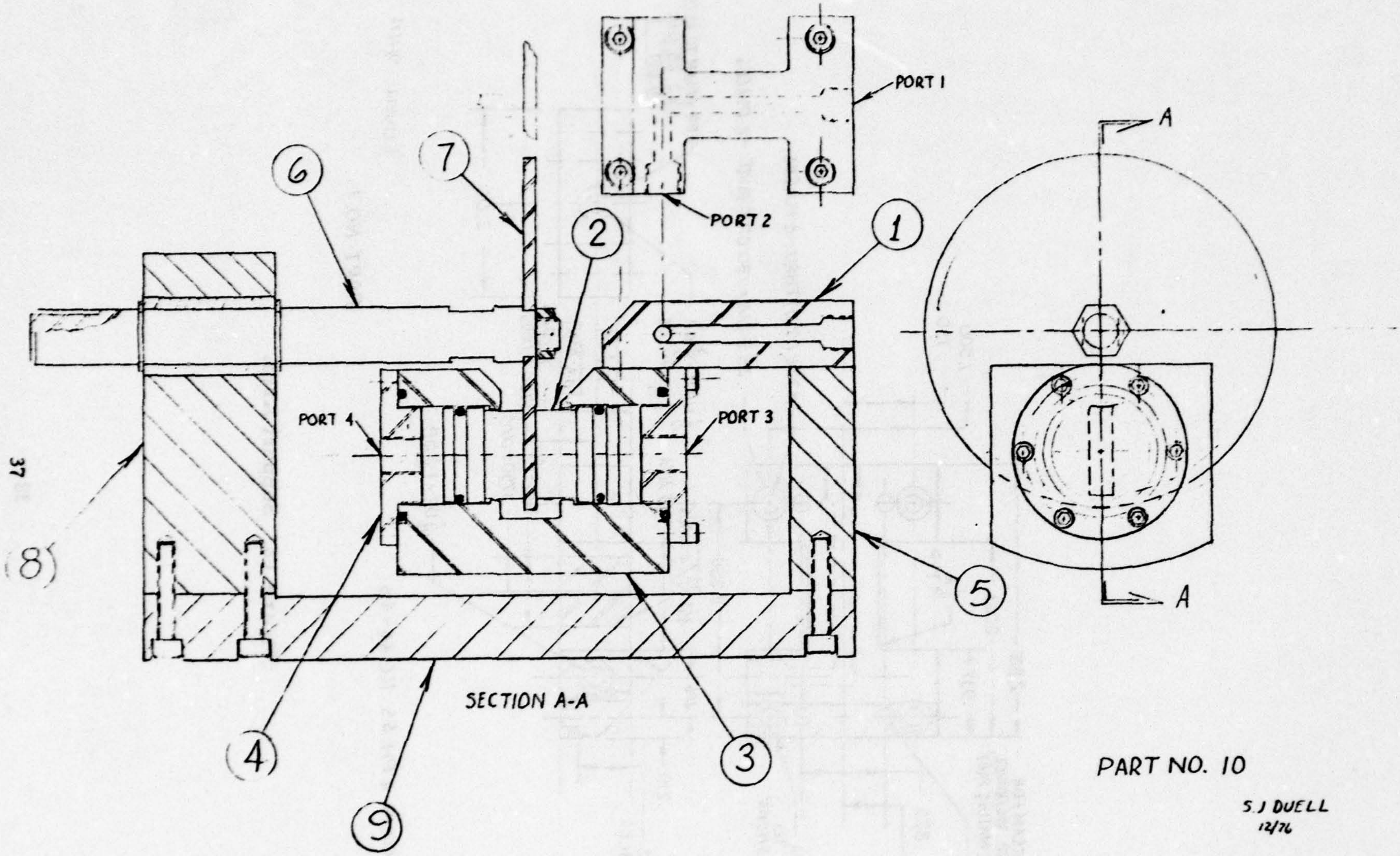


Figure 11. Assembly- Test Fixture for Solid Lubricant.

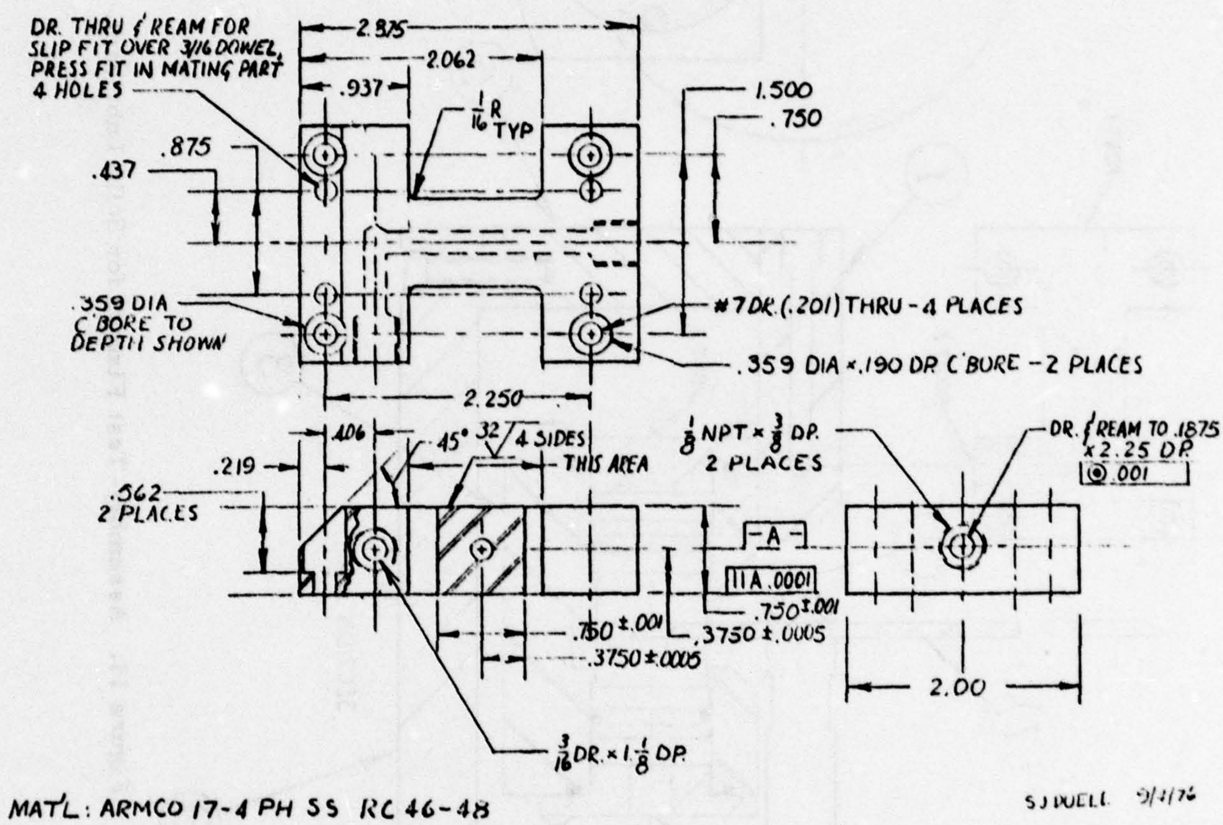


Figure 12. Support Arm.

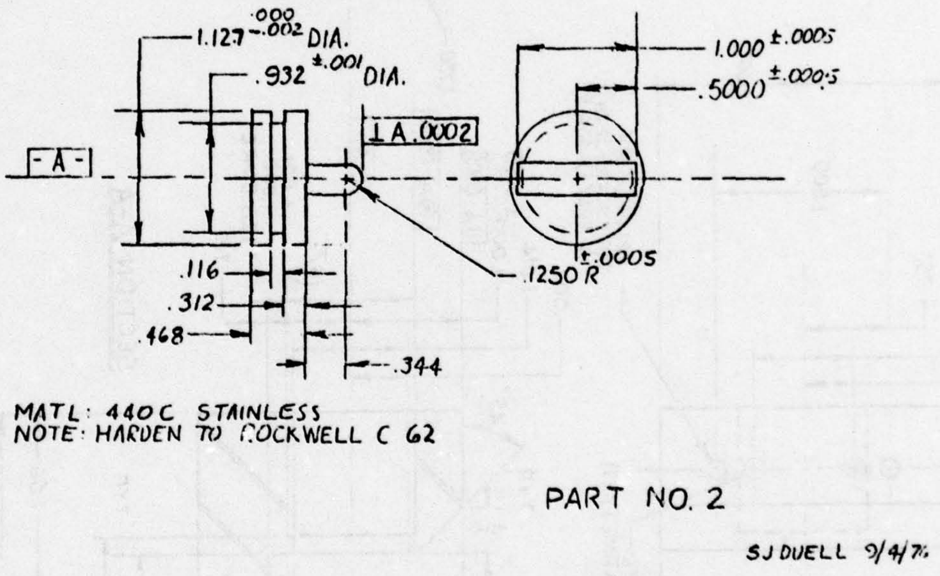


Figure 13. Piston (2 required).

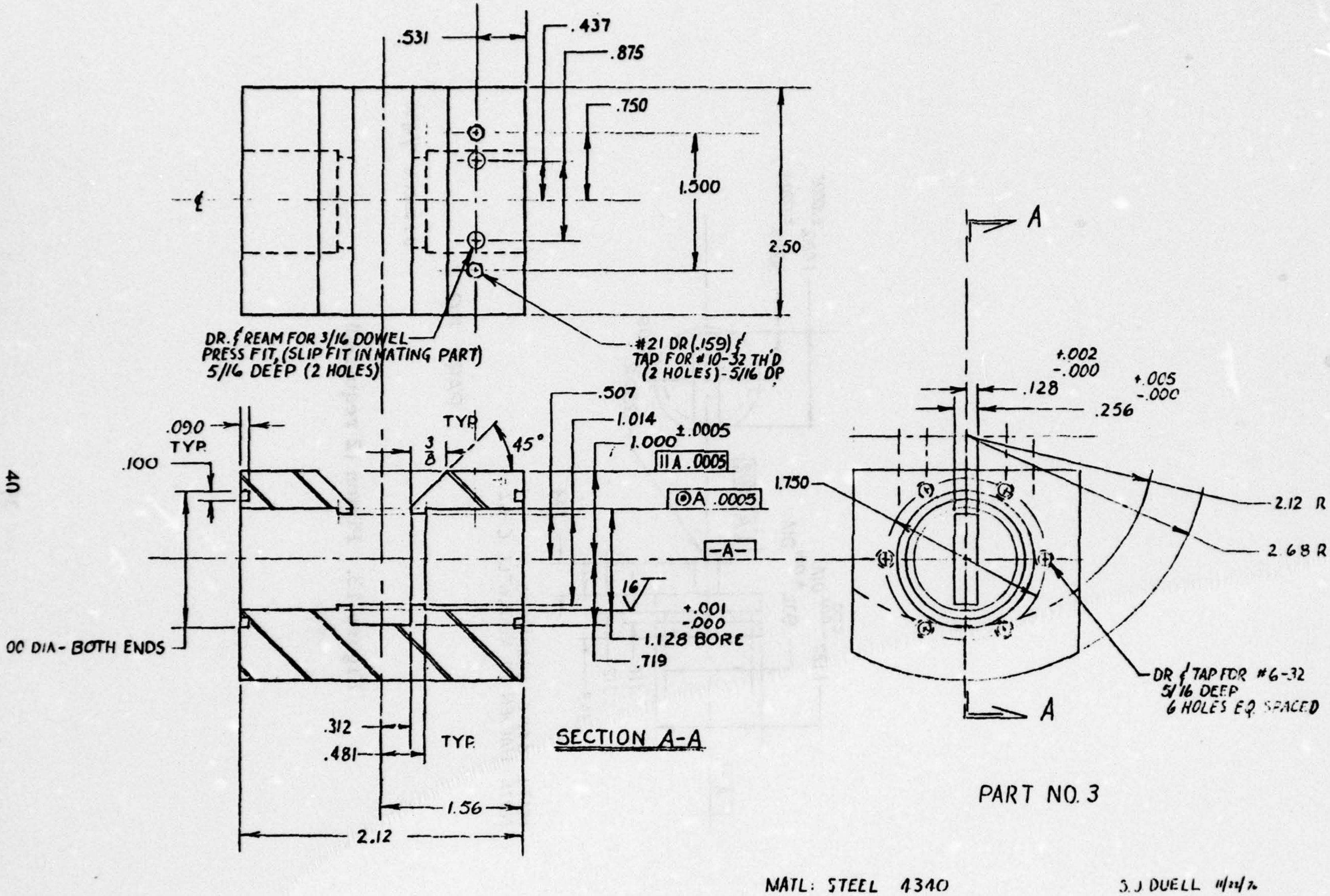


Figure 14. Cylinder.

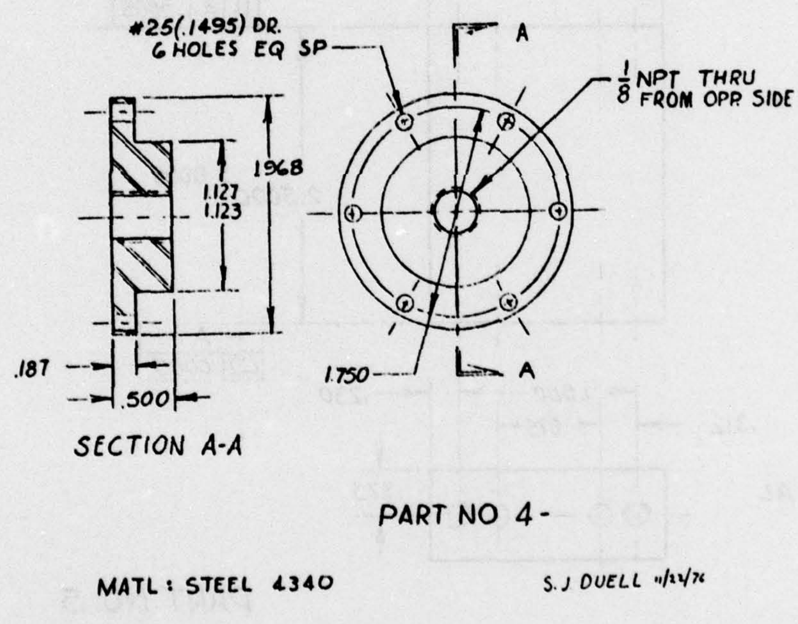


Figure 15. Cover (2 required).

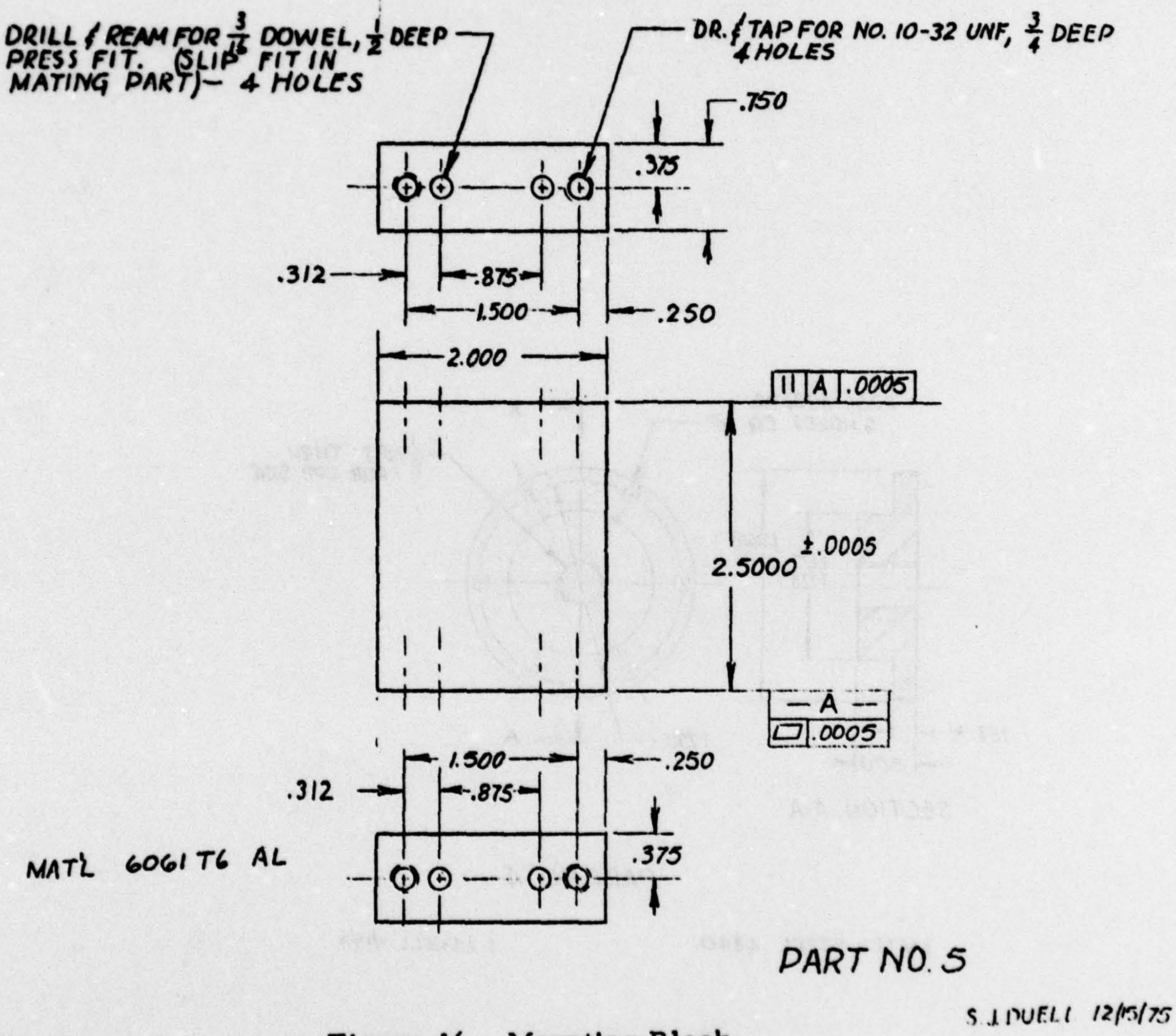


Figure 16. Mounting Block.

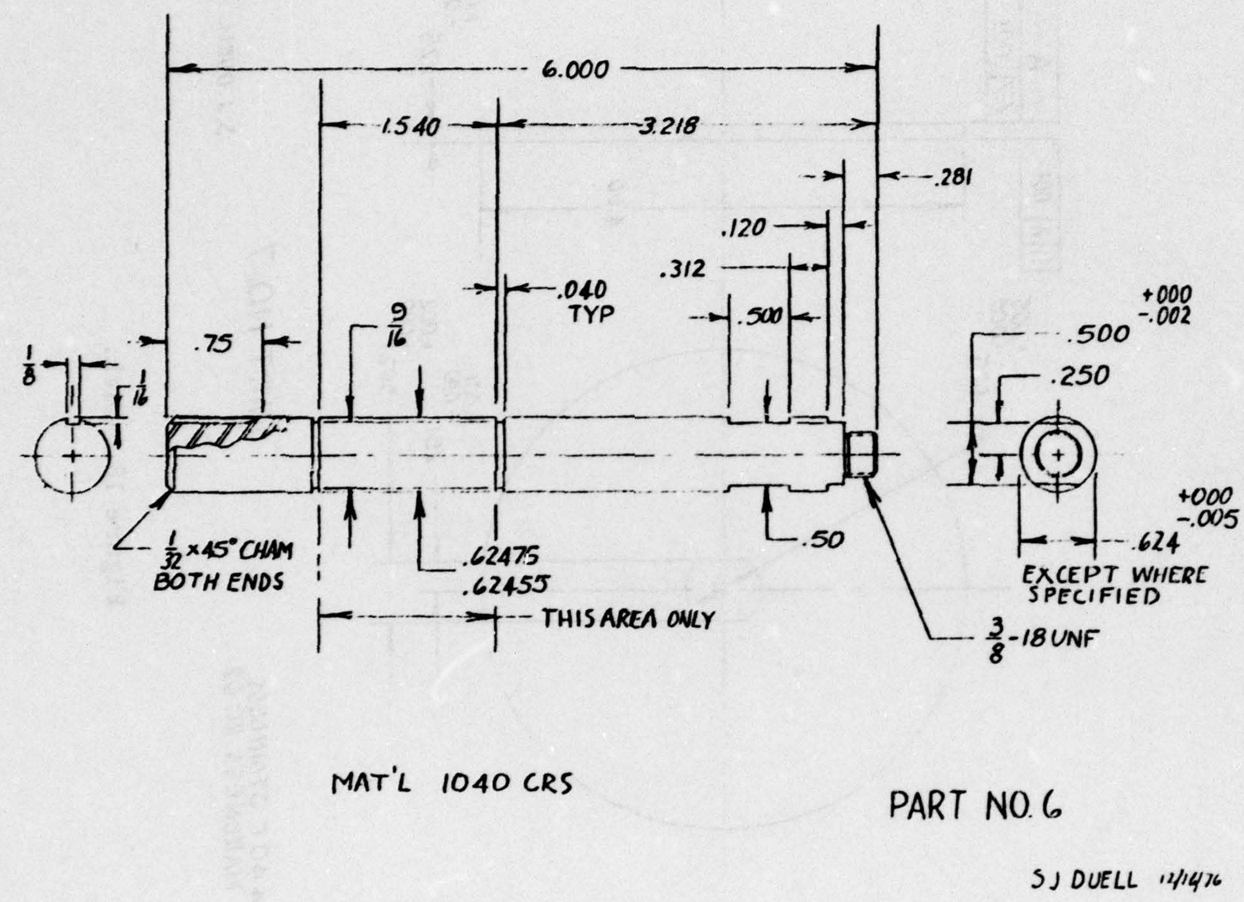


Figure 17. Shaft.

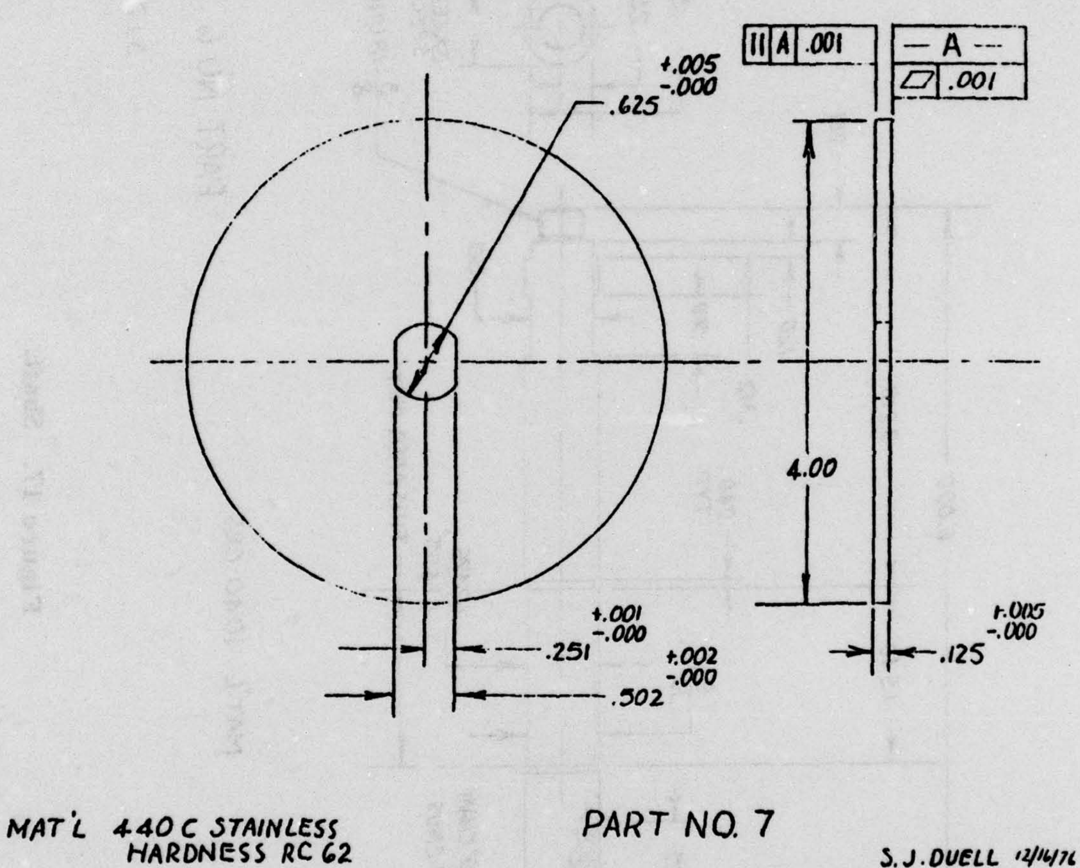


Figure 18. Disk.

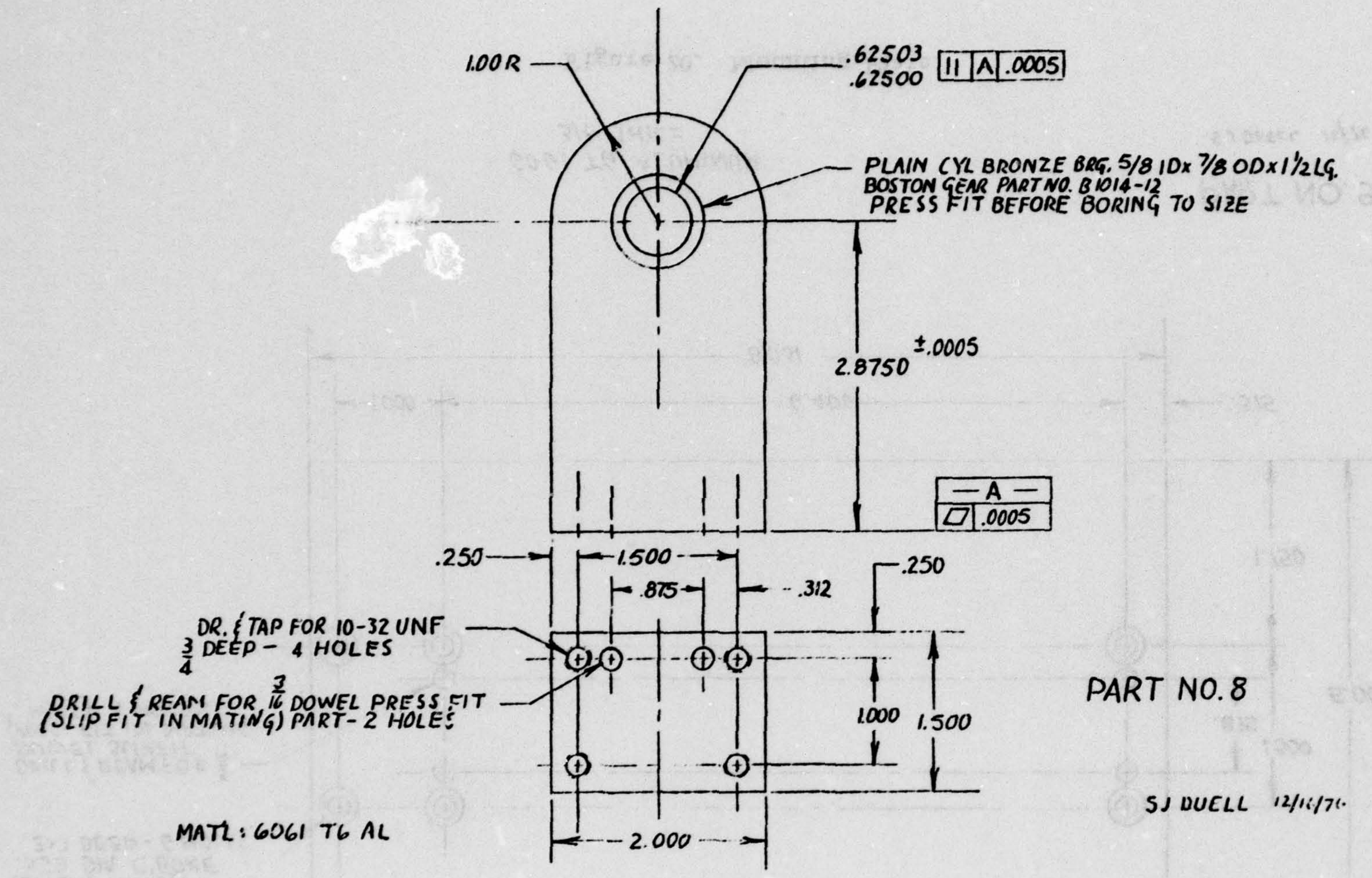


Figure 19. Bearing Mount.

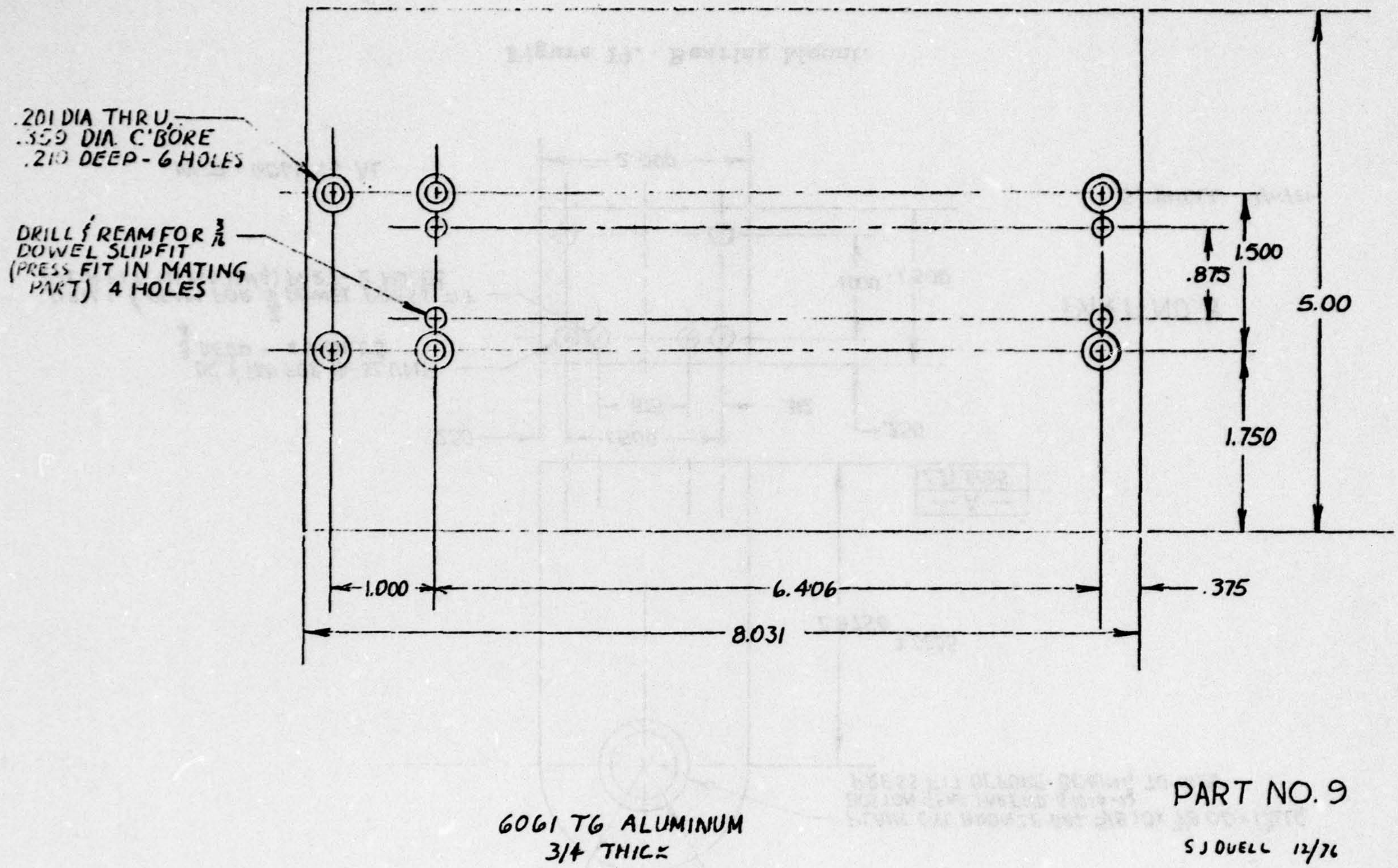


Figure 20. Mounting Plate

4.3.2 Operation

1. The disk will be coated on both sides with the test lubricant, and mounted on the shaft.

2. A variable speed motor (not shown) will be used to drive the disk at a speed which gives the desired surface speed.

3. For loading, hydraulic pressure will be applied by means of standard plumbing through port 1 of the support arm, and will be transmitted simultaneously to the pistons through ports 3 and 4 of the covers which are plumbed to port 2 of the support arm. Thus, balanced forces are applied to the two sides of the disk.

4. As the disk rotates under this load, a turning moment is exerted on the support arm.

a) By means of strain gages (not shown) mounted on the four sides of the square section of the support arm and wired in a bridge circuit to the appropriate circuitry, the *strain in the support arm due to the* torque can be sensed.

b) The strain gage transducer is to be calibrated so that the magnitude of the torque is known, depending on the output of the strain gage circuit.

5. The bore area of the cylinders is one square inch, thus giving a direct relationship between the hydraulic pressure and the load applied against the disk. Since the distance from the center of rotation to the center point of load application is known (1.375 in), the coefficient of friction between the disk and the nose of the piston can be found. In practice, the breakdown of a lubricant is assessed on the basis of a specified percentage increase in the coefficient of friction after a test run begins.

4.4 DESIGN DATA

4.4.1 Design Features

The design incorporates the features specified in the requirements. By using the opposed pistons, the need to provide a thrust bearing to handle the loading is eliminated. Perpendicularity, concentricity, and parallelism are tightly specified on various features of the components. Doweling of mating parts is used so that accurate assembly, once made, can be repeated should disassembly be required. Hydraulic sealing is done by means of o-rings. Replacement of worn pistons is easily accomplished by removing the covers and slipping the worn pistons out. With no pressure applied, the pistons can move back to give clearance for removal and installation of the disk. Flats on the shaft provide a wrench hold for tightening or removing the nut which retains the disk. The overall size is such that the apparatus can be readily mounted on a bench top or in an environmental chamber.

4.4.2 Hertzian Stress

Where a cylinder is loaded against a flat plate, the stress is given by the equation:[26]

$$S_c = 0.798 \sqrt{\frac{P/\ell}{D\left(\frac{-1-v1^2}{E_1} + \frac{1-v2^2}{E_2}\right)}} \qquad (1)$$

where,
- P = applied load, lb
- D = cylinder diameter, in
- E = modulus of elasticity, psi
- v = Poisson's ratio
- ℓ = cylinder length, in.

For this design,

$$v1 = v2 = 0.25,$$

$$E_1 = E_2 = 30 \times 10^6 \text{ psi},$$

$D = 0.25$ in.

$\ell = 1.0$ in.

Using these values in Equation (1), the loads required for various levels of stress are:

Load P, lb	Hertzian Stress, psi
247.0	100,000
353.0	120,000
981.5	200,000

Since the area of the cylinder bore is one square inch, the load P is simply the applied hydraulic pressure.

Equation (1) is, of course, applicable to elastic materials other than those specified in this design. The pistons and the disk are relatively simple parts to make, hence the design readily lends itself to the testing of solid lubricants on a variety of materials, or combinations of materials, which have moduli of elasticity of Poisson's ratios other than those specified here.

4.4.3 Design of the Support Arm

The material selected for the support arm is Armco 17-4 PH stainless steel, Rockwell C 46-48. Certain characteristics of this material make it preferable for the application of strain gages. A consideration in determining the dimensions of the arm is the level of stress which occurs at the surface when the bore through the arm is subjected to hydraulic pressure. This stress must be kept as low as practicable because the resulting strain is sensed by the strain gages along with the strain due to the torque. For calculating the stress at the surface, the arm was treated as a thick-walled cylinder. The appropriate equation[27] is:

$$\sigma_t = \frac{a^2 p_i}{b^2 - a^2} \left(1 + \frac{b^2}{r^2} \right) \tag{2}$$

where, σ_t = tangential stress, psi

a = inner radius, in

b = outer radius, in

r = radial position (R = b in this case), in

p_i = internal pressure, psi

Considering a possible pressure of 2500 psi, the section where the strain gages will be applied was designed to be 3/4 in square with a 3/16 in diameter bore. Applying Equation (2), the corresponding stress is:

σ_t = 333.3 psi.

With this stress level, the hydraulic pressure in the support arm will have negligible effect when the strain due to the torque is sensed.

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